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N, N, N, N-Tetramethylethylenediamine (TMEDA) and 1, 3-Diaminopropane (DAP) as Vapour Phase Corrosion Inhibitor (VPCI) for mild steel under Atmospheric conditions

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Abstract

Industrialization and modernization in now a days has made a strong demand of steel and their maintenance for a strong infrastructure for every country in the race of survival, stabilization, growth and competition. Atmospheric corrosion can aggressively accelerate the rate of degradation of steel during their manufacturing, processing, storage and transportation. In these cases, traditional methods to prevent corrosion are not suitable which provide scope of Vapour Phase Corrosion Inhibitors (VPCI) in industries, defense and daily life. N, N, N, N-Tetramethylethylenediamine (TMEDA) and 1, 3-Diaminopropane (DAP) were investigated as VPCI for mild steel under aggressive corrodents of atmosphere by Weight loss test, Eschke test, Salt spray test, Sulphur dioxide (SO₂) test at 50^oC and results of these tests were supported by Metallurgical research microscopy technique and SEM technique.

Keywords: Mild steel, weight loss test, Eschke test, salt spray test, metallurgical research microscopy, SEM, Vapour phase corrosion inhibitors.

Introduction

Atmospheric components such as moisture, air pollutants (SO_2 , H_2S , N_xO_y CO₂ and Cl⁻) and temperature have been reported as corrodents for metal corrosion. Corvo¹ and Moricelli et al.² studied the relationship between chloride concentrations with the corrosion rate in the atmospheric conditions. Ericsson³ showed that NaCl can cause corrosion at very low because it can induce corrosion by SO₂ on a carbon steel surface. NaCl can enhance 14 times rate of corrosion by SO2 at 9% relative humidity. In an another report of Blucher et al.⁴, they have investigated adverse effect of CO₂ on corrosion of Al. Vuorinen et al.⁵ and a list of authors have worked on the organic compounds as VPCIs. Due to presence of long chain hydrophobic part and the presence of atom having high electron density, organic compounds are the best selection for the compounds used as VPCIs. Organic substances have been studied as VPCI for mild steel were morpholine derivatives and diaminohexane derivatives⁵, fatty acid thiosemicarbazides⁶, cyclohexylamine and dicyclohexylamine⁷⁻⁸, amine carboxylates⁹, ammonium caprylate¹⁰, benzoic hydrazide derivatives¹¹⁻¹², polyamines¹³, bis-piperidiniummethyl-urea and β -amino alcoholic compounds¹⁴. Apart from organic substances, natural compounds like wood bark oil¹⁵ and thyme¹⁶⁻¹⁷ have also been used as VPCIs. Cano et al.¹⁸ recently have proposed mechanism of inhibition of dicyclohexamineisonitrite and dicyclohexaminenitrite against corrosion due to vapours of acetic acid and formic acid on carbon steel. Zubielewicz et al.¹⁹ studied the electrochemical behaviour of mixed anodic inhibitors. Batis et al.²⁰ evaluated the

performance the two primers, first natural rust converter and other on organic primer coating containing VPCI against atmospheric corrosion for reinforcing steel. Lyublinski²¹ studied synergistic corrosion management systems by use of corrosion inhibitors. In continuation to our earlier study²²⁻²⁷, in the present study, the inhibiting properties of N, N, N, N-Tetramethylethylenediamine (TMEDA) and 1, 3-Diaminopropane (DAP) were investigated on mild steel at 85 % relative humidity and 50 °C by Weight loss test, Salt spray test in a solution of 3.0 % NaCl, Eschke test, SO₂ test, Metallurgical research microscopy and Scanning electron microscopy.

Material and Methods

Many research papers, articles and reviews have been reported to the study of the techniques used to determine the effectiveness of VPCI against the metallic corrosion such as Adsorption isotherm technique²⁸, Weight loss technique²⁹, measurements³⁰. Potentiodynamic polarization meseaurment³¹, Electrodynamical impedance Audioradiography³² and Capacitance measurments³³. Tormoen et al.³⁴ reported three new techniques namely Surface-enhanced Raman spectroscopy, Scanning Kelvin probe microscopy and Contact angle analysis to monitor the adsorption of VPCI on the metallic surface in real time. These techniques can be used to evaluate the ability of two VPCI to diffuse and adsorb on the surface of metal simultaneously. Materials, equipments and methods used to investigate the amine as VPCI for mild steel at 50° C in my present study are explained as below:

Chemicals: N, N, N, N-Tetramethylethylenediamine (TMEDA): Minimum assay 99.0 %, Grade A.R., Source Spectrochem. Pvt. Ltd., Mumbai.

1, 3-Diaminopropane (DAP): Minimum assay 99.0 %, Grade A.R., Source Qualigens Fine Chemicals, Mumbai.

Mild Steel: ASTM-283, coupons of dimensions 3.5 cm \times 1.5 cm \times 0.025 cm and of percentage chemical composition: C – 0.17, Si – 0.35, Mn – 0.42, S – 0.05, P – 0.20, Ni – 0.01, Cu – 0.01, Cr – 0.01 and Fe - balance (w/w).

Along with them triply distilled water (conductivity $< 1 \times 10^{-6}$ ohm⁻¹ cm⁻¹) and sulphuric acid were also used.

Equipments: Weighing Balance: Single Pan Analytical Balance, Precision 0.01mg, Model AB 135-S/FACT, Source Mettler Toledo, Japan.

Humidity Chamber: Thermotech TIC-4000N Temperature Controller, Humidity controller with course and fine adjustments, AC Frequeny 50-60Hz, Max. Voltage 300V, Source Make-Associated Scientific Tech., New Delhi.

Salt Spray Chamber: Thermotech TIC-4000N Temperature Controller, Pumping system Pt-100, AC Frequeny 50-60Hz, Max. Voltage 300V, Source Make-Associated Scientific Tech., New Delhi.

Air Thermostat: Nine adjustable Chambered, Electrically controlled, Accuracy $\pm 0.1^{\circ}$ C.

Metallurgical Research Microscope: CXR II from Laomed, Mumbai, India.

Scanning Electron Microscope: JEOL 5900LV scanning electron microscope.

Methods: Vapour Pressure Determination Test: A definite amount of exactly weighed VPCI was placed in a single neck round bottom flask fitted with a rubber cork in the neck having a glass capillary of 1.0 mm diameter in the center of rubber cork. Then the flask was kept in electrically controlled air thermostat maintained at the constant temperature of 50^{0} C for 10 days. Change in weight of VPCIs was observed by analytical balance and vapour pressure of investigated VPCI was determined by weight loss of VPCI for time of exposure by equation 1.

$$\mathbf{P} = \begin{bmatrix} \mathbf{W} & \mathbf{2} \pi \mathbf{R} \mathbf{T} \\ \mathbf{M} & \mathbf{M} \\ \mathbf{M} & \mathbf{M} \end{bmatrix}$$
(1)

Where, P = vapour pressure of the VPCI (mmHg), A = area of the orifice (m²), t = time of exposure (sec.), W = weight loss of VPCI (kg), T = temperature (K), M = molecular mass of the inhibitor (kg) and R = gas constant (8.314 JK⁻¹mol⁻¹).

Weight Loss Test: Mild steel coupons were mechanically polished successively with the help of emery papers grading 100, 200, 300, 400 and 600µ and then thoroughly cleaned with plenty of triple distilled water, ethanol and acetone. Then coupons were dried with hot air blower and stored in desiccators over silica gel. Weight loss tests were carried out in an electronically controlled air thermostat maintained at a constant temperature of 50°C. After recording the initial weights of mild steel coupons, they were kept in different isolated chambers of air thermostat having fixed amount of VPCI at a constant temperature of 50° C for 24 hours of exposure time. A uniform thin film of VPCI was adsorbed onto the metal coupon after 24 hours of exposure. Then these coupons were transferred to a digitally controlled humidity chamber maintained at 85% humidity at a constant temperature of 50°C for 10 days. Blank coupons untreated with VPCI were also kept in the humidity chamber for the same duration in the same corrosive environment. After exposing the coupons for 10 days, coupons were taken out from the humidity chamber and washed initially under the running tap water. Loosely adhering corrosion products were removed with the help of rubber cork and coupon was again washed thoroughly with triple distilled water and dried with hot air blower and then weighed again. Corrosion rate in mils per year (mpy) and percentage corrosion inhibition efficiency (PCIE) were calculated by using the equations 2 and 3 respectively.

Corrosion Rate (mpy) =
$$\frac{534 \ X \ W}{DAT}$$
 (2)

Where, W = weight loss (in mg), D = density of mild steel (in g/cm^3), A = area of coupon (in sq. inch), T = exposure time (in hour).

Percentage Inhibition Efficiency =
$$\frac{CRo - CR}{CRo} \times 100$$
 (3)

Where, CRo = corrosion rate in absence of inhibitor and CR = corrosion rate in presence of inhibitor.

Salt Spray Test: After exposing the pre weighed mild steel coupons to VPCI in air thermostat for 24 hours, they were transferred to salt spray chamber having 3.0 % NaCl solution maintained at 50° C for duration of 10 days along with blank coupons. After exposing coupons for 10 days, coupons were treated in same manner as treated in weight loss test to remove corrosion products and then CR and PCIE were calculated.

Eschke Test: Kraft papers of suitable size were dipped in the VPCI for 30 seconds and then dried to adsorb uniform layer of inhibitor on Kraft papers. Mild steel coupons were wrapped in VPCI impregnated Kraft papers and then taken in humidity chamber maintained at 85 % relative humidity maintained at 50°C for first 12 hours and 25°C for next 12 hours alternately for 10 days. This temperature cycle was maintained in two sets because of formation and condensation of vapours of VPCI on mild steel surface regularly. After exposing coupons for 10 days, coupons were treated in same manner as treated in weight loss test to

remove corrosion products and then CR and PCIE were calculated.

Sulphurdioxide Test: SO₂ test was carried out on the mild steel coupons as in weight loss test. SO₂ gas was prepared by dissolving 0.04 g of sodium thiosulphate in 30mL aqueous solution of 1.0 % NH₄Cl and 1.0 % Na₂SO₄ solution and 0.5 mL of 1.0N H₂SO₄ was added to the flask. Initially pre-weighed and mechanically polished mild steel coupons were placed in air thermostat maintained at 50°C for duration of 10 days. Definite weight of VPCIs in a petridis and the flask, which is the source of SO₂, were placed in the isolated chambers of air thermostat containing mild steel coupons. After exposing coupons for 10 days, coupons were treated in same manner as treated in weight loss test to remove corrosion products and then CR and PCIE were calculated.

Metallurgical Research Microscopy: This test was employed to know about nature and type of corrosion using metallurgical research microscope. To investigate the corrosion inhibition efficiency of investigated VPCIs, micrographs of the corroded coupons treated with investigated VPCI were subjected to porosity study and morphology of surface. By the obtained results a comparative study of that porosity and surface morphology was carried which provided the information about the number of pores, size of pores, percentage porosity and area covered by the pores on the surface of coupon after the four different corrosion experiments. Percentage porosity (PP) and total objects (TO) shows the roughness of surface. On the other hand maximum

perimeter and maximum area object ratio (A/O) provide the information about the size and depth of the pores on the surface of mild steel. Micrographs of blank corroded coupons were taken after exposure of different aggressive environments for 10 days are shown in table-1 and results of metallurgical research microscopy of blank mild steel coupon after different corrosion tests are reported in table-2 from which it is clear that in weight loss test 9774 pores cover 8886066.4820 μ^2 area due to uniform corrosion in humid environment by which 68.90% surface become porous. In this test, numbers of pores are very high but A/O ratio is not very high as compared to that of salt spray test. In salt spray test, percentage porosity (69.94%) is almost equal to that of weight loss test but the numbers of pores (13,380) and the porous area (10960879.5014 μ^2) on the mild steel surface are high due to corrosive action of direct exposure of chloride ions on the surface of mild steel coupon. In this test, perimeter of pore (52323.4375μ) and A/O ratio are high due to large size and high depth of pores respectively. In SO₂ test, although the numbers of pores (3387) are very low as compared to other corrosion experiments yet the percentage porosity (86.52%) are highest in this test. In this test the size of pores (78541.5913 μ) and A/O ratio are very high due to high depth of the pores by the acidic action of SO₂ environment which provide evidence in favour of mechanism of pits formation on the surface of coupon by the acidic action of SO₂. In Eschke test, depth of pores is very low due to small size of pore of perimeter (20138.1682 μ) but total objects (6448) are high due to roughness of surface by the action of corrodents of environment.





Table-2	
Metallurgical research microscopy results of blank mild steel coupon in different corro	osion tests

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	ТО	PP	ΜΡ(μ)	$MA(\mu^2)$
Weight Loss Test	9774	68.9	55805.5407	8886066.4820
Salt Spray Test	13380	69.94	52323.4375	1096879.5014
SO ₂ Test	3783	86.62	78541.5913	9770443.2133
Eschke Test	6448	69.11	20138.1682	4461322.7147

Scanning Electron Microscopy: This technique gives the morphology study of mild steel coupons after treatment of different corrosion tests which provide the evidences in the support of inhibition data of investigated VPCIs, type of corrosion and for the mechanism of inhibition. In this test, samples were studied at different resolutions on the different spots on the mild steel coupons for complete information about the inhibition mechanism after treating with different tests. SEM of blank mild steel coupons were also taken for the comparative study of metal specimens which are given in figure-1. Micrographs of the blank coupons clearly provide the evidence of the pitting and crevice corrosion in corroding environments.



Figure-1 Scanning electron micrographs of blank mild steel coupon

Results and Discussion

Vapour Pressure Determination Test: Results of vapour pressure determination test for investigated VPCIs are given in table-3. Due to presence of sufficient vapour pressure, vapours of investigated VPCIs can easily adsorbed on the surface of mild steel coupon and form a barrier film for water vapours and corrosive aggressive contents of atmosphere around the coupons

and protect coupons from corrosion by the formation of protective layer.

 Table-3

 Vapour pressure of investigated VPCIs at 50°C

S. No.	Inhibitors	Vapour Pressure (mmHg)
1.	TMEDA	169.91 X 10 ⁻²
2.	DAP	178.92 X 10 ⁻²

Weight Loss Test: Results of CR and PCIE for investigated VPCIs obtained by weight loss test at 50°C are summarized in table-4. CR of mild steel treated with investigated VPCIs are given in figure-2 form which it is clear that the CR is very low on mild steel coupon which were treated with TMEDA and DAP. PCIE of investigated VPCIs are given in figure-3 from which it is clear that both investigated inhibitors work well against corrosion on the mild steel under atmospheric conditions at high temperature.

Table-4 Weight loss test parameters obtained for VPCIs at 50°C and 85% relative humidity for 10 days

S.No.	VPCI	Weight Loss(mg)	CR (mpy)	PCIE
1.	Blank	14.8	5.12	-
2.	TMEDA	4.1	1.41	72.35
3.	DAP	3.4	1.17	77.05

Figure-2

Corrosion rate of mild steel coupon treated with VPCI with



respect to blank coupons obtained from Weight loss test

Salt Spray Test: Results of weight loss and CR of mild steel after 10 days exposure of NaCl at 50°C are shown in figure-4. It is clear from figure-4 that in salt spray test, direct contact of chloride ions on mild steel coupons accelerate the rate of corrosion. From PCIE of VPCIs in figure-5, it is shown that both investigated

VPCIs were worked very well against the salt spray at high temperature.



Figure-3 Percentage corrosion inhibition efficiency of TMEDA and DAP obtained from Weight loss test



Figure-4 Weight loss and corrosion rate of mild steel coupon treated with VPCIs obtained from Salt spray test



Figure-5 Percentage corrosion inhibition efficiency of TMEDA and DAP obtained from Salt spray test

Eschke Test: Weight loss of the mild steel coupons, CR and PCIE of VPCIs were calculated at 50°C for duration of 10 days in Eschke test and the data obtained are shown in figure-6 and figure-7. It is clear from the figure-6 that both VPCIs perform very significant role against the corrosion at high temperature. Visual observations of mild steel coupons are also given in table-5.



Figure-6

Weight loss and corrosion rate of mild steel coupon treated with VPCIs obtained from Eschke test



Figure-7 Percentage corrosion inhibition efficiency of TMEDA and DAP obtained from Eschke test

SO₂ Test: Results obtained for different corrosion determination parameters by SO₂ test are given in figure-8 and figure-9. It is seen that value of CR of mild steel coupons treated with VPCIs is high in this test for the same duration as compared with CR in Eschke test due to the acidic environment of SO₂. From figure-9 it is shown that both investigated VPCIs play significant role to prevent the mild steel from the corrosion in acidic environment of SO₂ which is part of atmospheric gases near the industries.



Figure-8

Weight loss and corrosion rate of mild steel coupon treated with VPCIs obtained from SO₂ test.



Figure-9 Percentage corrosion inhibition efficiency of TMEDA and DAP obtained from SO₂ test

Metallurgical Research Microscopy: Metallurgical Research Microscopy of TMEDA: Results of metallurgical research microscopy and micrographs of mild steel coupons treated with TMEDA after corrosion tests are reported in table-6 and table-7 respectively. By comparison of the data obtained by different corrosion tests, it is clear that percentage porosity (PP) is significantly low in all tests due to good inhibition action of TMEDA against the atmospheric corrosion. It is shown that PP is almost negligible in Eschke test (3.14%) due to direct contact of TMEDA with surface of mild steel coupon. In weight loss test, 2119 pores cover 5346.2604 μ^2 area due to uniform corrosion in humid environment by which 8.29% surface become porous. In salt spray test, PP (9.95%) is not very high but the numbers of pores (4645) are very high due to more roughness by direct exposure of NaCl along with water vapours on the surface of mild steel coupon. Small porous area of mild steel coupon in salt sprat test is due to inhibition action of TMEDA against pitting and crevice formation. In this test, perimeter of pore (4399.6332 μ) and A/O ratio are very high due to large size and high depth of pores respectively. In SO₂ test, although numbers of pores (1625) are not very high yet its A/O ratio is very high due to very large pores having high

depth. In Eschke test, depth of pores are negligible due to small size of pore of perimeter 513.0056 μ but total objects (1739) are higher than pores in SO₂ test due to roughness on surface of mild steel by the action of corrodents of environment. Micrographs of all tests show that almost smooth surface of mild steel coupon without any corrosion products are obtained after the treatment of coupon with TMEDA.

Metallurgical Research Microscopy of DAP: Results of metallurgical research microscopy and micrographs of mild steel coupon treated with DAP after four different corrosion tests are reported in table-8 and table-9 respectively. In weight loss test, 3613 pores cover 9612.1884 μ^2 area due to uniform corrosion in humid environment by which 7.38% surface become porous. In salt spray test, PP (10.68%) and numbers of pores (6215) are some high due to direct spray of salt on surface of coupon. In this test, perimeter of pore (8598.8847 μ) are little bit high but A/O ratio are significantly low due to corrosion inhibition action of DAP which prevent the formation of pits on the surface of mild steel coupon by the action of chloride ion. In SO_2 test, although numbers of pores (1941) are low as compare to weight loss test and salt spray test yet its A/O ratio is high due to pores having high depth because the perimeters of pores (6825.0334 u) are also low than that of salt sprav test. In Eschke test, depth of pores is comparatively low due to small size of pore of low perimeter (1958.7325 µ) but total objects (3787) are high due to roughness of surface by the action of corrodents of environment. Micrographs of all tests show that very smooth surface of mild steel coupon without any corrosion products are obtained after the treatment of coupon with DAP.

Scanning Electron Microscopy: By comparison of SEM images of coupons treated with TMEDA and DAP as shown in table-10, it is clear that both VPCIs show very excellent corrosion inhibition properties against the aggressive environments. SEM image of TMEDA have no any corrosion product and no pits even at very high resolution of 15000. Clearness of SEM image of coupon treated with DAP provide the evidence in favor of very good PCIE of DAP.

VPCI Salt Spray Test			
		Escnke Test	SO ₂ Test
Blank	Clear pits and crevices were visible	Uniform corrosion	Pitting corrosion
TMEDA	Clean surface No corrosion product	Clean surface No corrosion product	Clean surface No corrosion product
DAP	Slightly tarnishing No any pits and crevice No corrosion product	Clear clean surface No corrosion product	Slightly tarnishing No any pits and crevice No corrosion product

 Table-5

 Visual observations of mild steel coupons surface after performed various tests

 Table- 6

 Micrographs of mild steel coupons treated with TMEDA in different corrosion tests

Micrograph of Wt. Loss Test	Micrograph of Salt Spray Test
Micrograph of SO ₂ Test	Micrograph of Eschke Test

Table-7

Total objects (TO), percentage porosity (PP), maximum perimeter (MP) of pore and maximum area (MA) covered by pore on mild steel coupon after treated with TMEDA after different corrosion tests

	ТО	PP	ΜΡ(μ)	$MA(\mu^2)$
Weight Loss Test	2119	8.29	3604.6711	15346.2604
Salt Spray Test	4645	9.95	4399.6332	24806.0942
SO ₂ Test	1652	10.86	5889.6992	25318.5596
Eschke Test	1739	3.14	513.0056	1066.4820

 Table-8

 Micrographs of mild steel coupons treated with DAP in different corrosion tests

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Micrograph of Wt. Loss Test	Micrograph of Salt Spray Test
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Micrograph of SO ₂ Test	Micrograph of Eschke Test

Table-9

Total objects (TO), percentage porosity (PP), maximum perimeter (MP) of pore and maximum area (MA) covered by pore on mild steel coupon after treated with DAP after different corrosion tests

	ТО	PP	ΜΡ (μ)	ΜΑ (μ²)
Weight Loss Test	3613	7.38	2855.8658	9612.1884
Salt Spray Test	6215	10.68	8598.8847	18664.8199
SO ₂ Test	1941	8.5	6825.0334	14325.8476
Eschke Test	3787	6.44	1058.7325	6908.5873

 Table-10

 SEM image of mild steel coupons treated with TMEDA and DAP after corrosion test



Mechanism of Inhibition: A deep analysis of the results reveals that the probable mechanism of inhibition action of investigated VPCIs contains the following features: i. presence of lone pair donar N atoms in the molecule of TMEDA and DAP provide them specific active functional groups by which it can be easily adhere on the surface of mild steel due to acid-base nature of steel and VPCI respectively. ii. due to high vapour pressure it saturates the environment around the mild steel and excludes the corrosive contents. iii. presence of four methyl groups directly attached with the lone pair donar atom in TMEDA enhance the basic strength of TMEDA molecule due to inductive effect by which it can easily neutralized the acidic environment around the mild steel.

Conclusion

As a result of experimental work carried out on the performance of investigated vapour phase corrosion inhibitors, a deep analysis of corrosion parameters obtained by corrosion testing experiments, morphology of mild steel coupon show that both investigated VPCIs (TMEDA and DAP) perform excellent corrosion inhibition properties against the aggressive environments of SO₂ and NaCl at high relative humidity and high temperature. Due to basic nature and high vapour pressure, their vapours neutralize the acidity of atmospheric environment around mild steel and provide barrier for corrosive contents. In Eschke test and Salt spray test, the order of PCIE of TMEDA in comparison of DAP is exactly reversed in Weight loss test and SO₂ test due to high basic properties and surface area of TMEDA molecule. Metallurgical research microscopy and Scanning electron microscopy give an idea of the uniform type, crevice type and pitting type of corrosion on mild steel in humid environment, NaCl environment and in SO₂ environment respectively.

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