

Study of Vapour Phase Corrosion Inhibitors for Mild Steel under different Atmospheric Conditions

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Abstract: Mild steel is used as main raw material in fabrication of equipment, chief material of construction and as weapons. During the storage and transportation conditions, it comes in contact with aggressive environment which leads to decrease in mechanical strength. Four different vapor phase corrosion inhibitors (VPCIs) i.e. *n*-Caprylic acid (CA), *n*-Butyric acid (BA), *N,N*-dimethyl propylene urea (DMPU) and 2-Amino Benzothiazole (ABT) were tested under different atmospheric conditions at 40 °C by Weight Loss, Eschke test, Salt Spray and SEM techniques. All the four VPCIs show very high corrosion inhibition efficiency i.e. 79-99 %. The results obtained from corrosion experiments were supported by SEM images.

Keywords: Mild steel, Atmospheric corrosion, Vapor phase corrosion inhibitors, Eschke Test.

I. INTRODUCTION

Mild steel is the most common form of steel and because of its low cost it is chief material of construction. Mild steel have good strength, hard and can be bent, worked or can be welded into an endless variety of shapes for uses from vehicles (like cars and ships) to building materials. Because of its unique properties like, very cheap, high strength, hardness and easy availability, it has wide range of applications in nut bolt, chains, hinges, knives, armour, pipes, magnets, military equipments etc. Atmospheric corrosion also known as vapor phase corrosion (VPC) is due to the individual and combined action of oxygen, moisture, and atmospheric pollutants. Additional contributors to VPC are rain, snow, dust, soot, ash, wind, and radiation (light, heat, etc.). The rate of VPC may be accelerated by both acids and bases, depending upon the metal and their alloys are exposed to aggressive environment under atmospheric condition during the manufacture, processing, storage, or transportation and can accelerate the degradation of the metal, alloys and their products. In such cases, the corrosion prevention methods like water-displacing products (oil or grease), water-absorption products (silica gel) and dehumidification are not significant due to high labor, material cost for the application and removal of product and difficulty to calculate specific moisture. The vapor phase corrosion inhibitors (VPCI) play a significant role in minimizing corrosion to metals and their alloy in atmospheric condition by producing vapors with sufficient vapor pressure due to their volatile nature, and prevent the metal or alloys from corrosion by adsorption of their vapors onto the metal surface.

The effective use of surfactants for VPCI depends upon environment and properties of metals as well as surfactants [1-5]. It has been shown for iron that when relative humidity is below 60 percent, no corrosion is expected, while above 75 to 80 percent, the degree of corrosion will be high [6]. Detrimental effects caused by photochemical reactions of sulphur dioxide and nitrogen oxides have been demonstrated by Stern [7]. Use of VPCI is an effective method to prevent atmospheric corrosion [8, 9]. The corrosion inhibitors volatilizes into the atmosphere surrounding the metal and thus modify the atmosphere [10].

The choice of a chemical compound as VPCIs depends upon on its vapor pressure and efficiency to prevent corrosion by forming a protective film. The vapor pressure of VPCI must possess some optimum values. If the vapor pressure of the VPCI is too low, e.g. in the order of 10^{-6} Torr at room temperature, the protective vapor concentration will be established only slowly. This may result in insufficient corrosion protection during an initial time period. Further, if the space that houses both the stored equipment and VPCI is not sealed, sufficient inhibitor concentration may not be reached. Conversely, under similar conditions, if the vapor pressure of VPCI is too high (approx. 0.1 Torr at ambient conditions), its effectiveness will be limited to a short time period, as its consumption rate will be high.

Subramanian et. al [11] studied the most commonly used VPCI, derivatives of ammonium carbonate and ammonium nitrite on copper, mild steel and zinc in sulphur dioxide (SO₂) environments. Due to their easily availability and their better percentage corrosion inhibition efficiency (PCIE) they have been used in industry for several decades. However, the disadvantages of using these derivatives are their toxic nature to the environment. Thus, replacing them with new environmental friendly inhibitors is desirable.

Saurbier et. al [12] suggested toluylalanine as an effective temporary inhibitor of steel in wet atmosphere. Vuorinew [13] reported a series of morpholine-mannich based derivatives as volatile corrosion inhibitors. Polymeric corrosion inhibitors such as polyacrylic and polyamino-benzoquinone etc. are widely used and they have a lower toxicity than their monomers [14, 15]. Many kinds of morpholine oligomer (MPO) as VPI for the temporary protection of box shaped hatch covers and

rudder blades of large ships at Hudong Shipyard have been studied by Zhang *et al.* [16].

Quraishi *et al.* [17] studied the inhibiting properties of five organic vapor phase inhibitors namely, derivatives of imidazoline maleate, orthophosphate, nitro benzoate, phthalate, cinnamate on mild steel, brass and copper. They also studied some organic volatile corrosion inhibitors mostly derivative of diaminohexane such as diaminohexane cinnamate, nitro benzoate, phthalate, orthophosphate and maleate on aluminum, zinc and mild steel [18].

Study of some salts of benzoic hydrazide benzoate (BHB), benzoic hydrazide salicylate (BHS) and benzoic hydrazide nitro benzoate (BHN) as corrosion inhibitors of mild steel [19-21], brass and copper was studied by weight loss method [22] (Quraishi, 2005). Persiantsava, examined derivatives of benzene with β -naphthol as a VPCI in a sulphur dioxide and chloride atmosphere [23]. Subramanian *et al.*, studied the corrosion inhibition behavior of morpholine and its three derivatives salts-morpholine carbonate, borates, and phosphates salts [24]. Of these morpholine and its carbonates salt exhibited 90 and 85% corrosion inhibition efficiency (CIE) respectively while the other salts gave less than 40% corrosion inhibition efficiency.

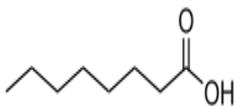
In the present study, the inhibiting properties of four organic VPCIs named as n-Caprylic acid (CA), n-Butyric acid (BA), N,N-dimethyl propylene urea (DMPU) and 2-Amino Benzothiazole (ABT) were investigated on mild steel by Weight Loss technique at 85 % of relative humidity and 40 °C temperature, Salt Spray method in a medium of 3.0 % sodium chloride, Eschke test and SEM techniques.

II. EXPERIMENTAL WORK

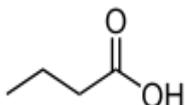
Name, structure and molecular formula of four VPCI for Mild steel are shown in Table 1. These VPCIs were selected due to their easily availability, suitable vapor pressure, less toxic nature, high durability, and cost effective nature.

Table 1. Name and structure of four Vapour Phase Corrosion Inhibitors

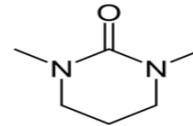
- i) n-Caprylic acid (CA) :
Mol. Weight 144.21 g/mol



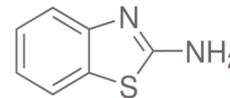
- ii) n-Butyric acid (BA) :
Mol. Weight 88.11 g/mol



- iii) N,N-dimethyl propylene urea (DMPU):
Mol. Weight 128.17 g/mol



- iv) 2-Amino Benzothiazole (ABT):
Mol. Weight 150.2 g/mol



Mild steel (ASTM-283) used for the investigation was in the form of sheet (0.025 cm thick) and had the following 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). The mild steel coupons of dimensions 3.0 cm × 1.5 cm × 0.025 cm were used for different corrosion tests. Metal specimens were mechanically polished successively with the help of emery papers of grades 100, 200, 300, 400 and 600 micron and then thoroughly cleaned with plenty of triple distilled water (conductivity less than 1×10^{-6} ohm⁻¹ cm⁻¹) and then with acetone. The specimens were dried with hot air blower and stored in desiccators over silica gel. Duplicate in some case triplicate experiment were performed to check the reproducibility of data.

A. Vapor Pressure Determination Test:

A standard Knudsen method [25] was used to determine the vapor pressure of all the four VPCIs. Definite amount of exactly weighed VPCIs were 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 air thermostat maintained at the constant temperature of 40 °C for 10 days. Change in the weight of VPCIs was observed by the single pan analytical balance (0.01 mg accuracy). Vapor pressure of all the four investigated VPCIs was determined by equation (1) and has been shown in Table 2.

$$P = \frac{W}{At} \left[\frac{2\pi RT}{M} \right]^{1/2} \quad \dots(1)$$

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

Table 2. Vapour pressure of all the four investigated Vapour Phase Corrosion Inhibitors.

S. No.	Corrosion Inhibitors	Vapour pressure (mm Hg)
1.	N-Caprylic acid	5.31

2.	N-butyric acid	4.58
3.	DMPU	2.10
4.	2-Amino Benzothiazole	2.116

B. Weight Loss Technique

Weight loss experiments were carried out in an electronically controlled air thermostat (perfectly insulated) maintained at a constant temperature of 40 °C with in an accuracy of ±0.1°C. Four vapor phase corrosion inhibitors namely N-N-dimethyl aniline (DMA), Morpholine, Cyclohexyl amine (CHA) and Hexamethylene imine (HMI) were placed separately in different isolated chamber in the specially designed air thermostat. After recording the initial weights of mild steel specimens on a Mettler Toledo, Japan AB 135-S/FACT, single pan analytical balance, (with a precision of 0.01 mg), they were kept in different isolated chamber (perfectly insulated from each other) of air thermostat having fixed amount of VPCI at a constant temperature of 40 °C for 24 hours of exposure time. A uniform thin film of VPCI was adsorbed onto the metal coupons after 24 hours of exposure. Then these coupons were transferred to a digitally controlled humidity chamber maintained at 85.0 % humidity at a constant temperature of 40 °C for 10 days. Blank coupons were also kept in the humidity chamber for the same duration in the same corrosive environment. After exposing the specimens for 10 days, the specimens 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 the specimen was again washed thoroughly with triple distilled water and dried with hot air blower and then weighed again. Corrosion rates (CR) in mils per year (mpy) and percentage corrosion inhibition efficiency (PCIE) were calculated using the equations (2) and (3) respectively [26].

$$\text{Corrosion rate (mpy)} = \frac{534 \times W}{DAT} \quad \dots(2)$$

Where, W = Weight loss (mg), D = Density of mild steel (gm/cm³), A = Area of specimen (sq. inch), T = Exposure time (hours).

$$\text{PCIE} = \frac{CR_{Blank} - CR_{inhibitor}}{CR_{Blank}} \times 100 \quad \dots(3)$$

Where, CR_{Blank} = corrosion rate in blank and CR_{inhibitor} = corrosion rate in presence of inhibitor.

C. Salt Spray Method

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 % sodium chloride solution maintained at constant temperature of 40 °C for duration of 10 days along with blank specimens. After exposing the specimens for 10 days, the specimens were

taken out from the salt spray chamber and washed initially under the running tap water. Loosely adhering corrosion products were removed with the help of rubber cork and the specimen was again washed thoroughly with triple distilled water and dried with hot air blower and then weighed again. Corrosion rate (mpy) and PCIE were calculated using the equations (2) and (3), respectively.

D. Eschke Test

Eschke test was carried out on the pre weighed mechanically polished mild steel coupons as prescribed in the literature [27]. Kraft papers of suitable size were dipped in the VPCI for 30 second and then dried to adsorb uniform layer of the inhibitor on the Kraft papers. Then mild steel coupons were wrapped in VPCI impregnated Kraft papers and then transferred into the humidity chamber maintained at 85% relative humidity maintained at a constant temperature of 40°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 the vapors of VPCI on mild steel surface regularly. After exposing the specimens for 10 days, the specimens 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 the specimen was again washed thoroughly with triple distilled water and dried with hot air blower and then weighed again. Corrosion rate (mpy) and PCIE were calculated using the equations (2) and (3), respectively.

E. Scanning Electron Microscopy (SEM) Technique

This technique is employed for the surface study of mild steel coupons to know about nature and type of corrosion using SEM technique. The micrographs of the corroded specimens were taken after exposure of 10 days. Micrographs of the blank mild steel coupons were also taken for the comparative study of metal specimen.

III. RESULTS AND DISCUSSIONS

A. Weight Loss Technique

The values of weight loss (mg), corrosion rate (mpy) and PCIE for all the four VPCIs were shown in Table 3. The corrosion rate is found to be almost negligible in the coupons of mild steel treated with CA and BA. PCIE of all the four investigated VPCIs are shown in Fig. 1.

Table 3. Weight loss (mg), CR (mpy) and PCIE for all the four VPCIs for mild steel at 40 °C and 85.0% relative humidity after 10 days of exposure by weight loss method.

S. No.	VPCI	W.Loss (mg)	CR (mpy)	PCIE
1.	Blank	61.45	25.195	-
2.	N-Caprylic acid	0.43	0.176	99.30
3.	N-butyric acid	4.02	1.648	93.45

4.	DMPU	12.75	5.227	79.25
5.	2-Amino Benzothiazole	12.17	4.98	80.23

PCIE follows the same order as in weight loss method i.e. CA > BA > ABT > DMPU.

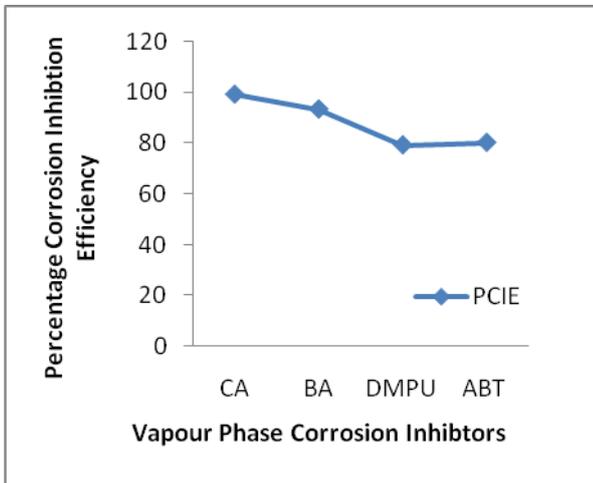


Fig 1. Percentage corrosion inhibition efficiency of all the four vapor phase corrosion inhibitors by weight loss method

It is clear from Fig. 1 that all the four investigated VPCIs shows high PCIE i.e. 80-99 %. Out of the four investigated VPCIs, CA exhibit highest PCIE i.e. 99.3% for the mild steel at 40.0 °C and DMPU shows minimum i.e. 79.25. PCIE follows the order as CA > BA > ABT > DMPU.

B. Salt Spray Method

Weight loss (mg), corrosion rate (mpy) and PCIE of all the four investigated VPCIs at a temperature of 40.0 °C by Salt Spray method were shown in Table 4.

Table 4. Weight loss (mg), CR (mpy) and PCIE of all the four VPCIs for mild steel at 40 °C and 85.0 % Relative humidity after 10 days of exposure by Salt Spray Method.

S. No.	VPCI	Weight loss (mg)	CR (mpy)	PCIE
1.	Blank	85.47	35.49	-
2.	N-Caprylic acid	7.21	2.99	91.57
3.	N-butyric acid	16.32	6.77	80.92
4.	DMPU	25.67	10.66	69.96
5.	ABT	25.12	10.29	71.00

Fig. 2 shows percentage corrosion inhibition efficiency of all the four investigated VPCIs. As chloride ions are very aggressive from corrosion point of view, so a high corrosion rate was observed in salt spray method in comparison to weight loss method. All the four investigated VPCIs shows good corrosion inhibition efficiency i.e. 71.0 to 91.0 % even in this aggressive environment and at a high temperature of 40.0 °C. The

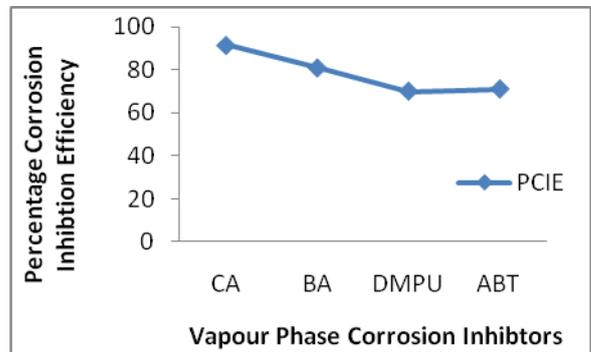


Fig 2. Percentage corrosion inhibition efficiency of all the four vapor phase corrosion inhibitors by Salt Spray method

C. Eschke Test

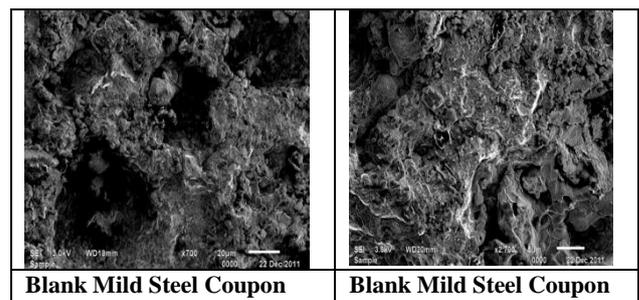
Weight loss (mg), corrosion rate (mpy) and PCIE of all the four VPCIs at 40.0 °C after 10 days of exposure by Eschke test were shown in Table 5. Fig. 3 shows PCIE of all the four investigated vapor phase corrosion inhibitors. It is clear from the Fig. 3 that all the four investigated VPCIs shows very high PCIE i.e. more than 87.0 %. Out of these four, BA shows 98.38 PCIE for mild steel. The PCIE follows the order i.e. BA > CA > ABT > DMPU.

Table 5. Weight loss (mg), CR (mpy) and PCIE of all the seven investigated VPCIs for mild steel at 40.0 °C and 85.0 % relative humidity after 10 days of Exposure by Eschke Test.

S. No.	VPCI	Weight loss (mg)	CR (mpy)	PCIE
1.	Blank	41.42	17.18	-
2.	N-Caprylic acid	5.29	2.169	87.37
3.	N-butyric acid	0.68	0.278	98.38
4.	DMPU	3.55	1.455	91.53
5.	ABT	4.12	1.68	90.22

D. SEM technique

SEM images of mild steel coupons treated with different VPCIs by weight loss method after exposure of 10 days at 40.0 °C were shown in Fig. 4.



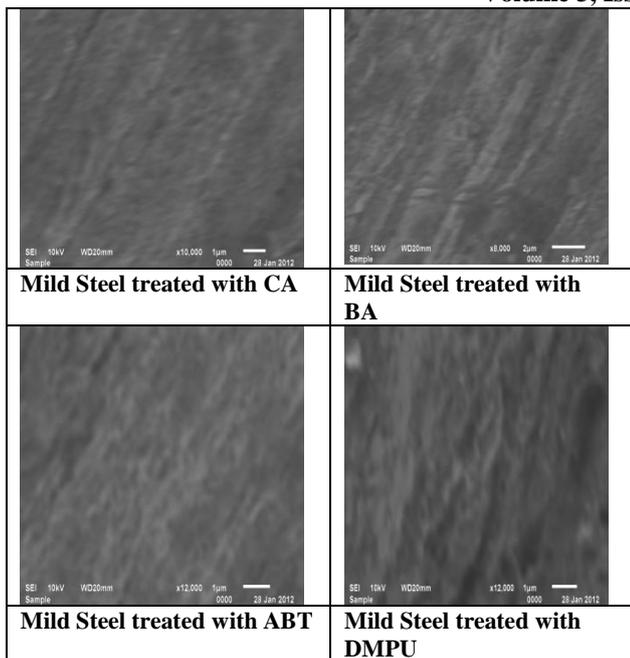


Fig 4. SEM images of mild steel coupons blank and treated with VPCIs

Pits are clearly visible in the images of blank coupons of mild steel showing pitting type of corrosion in absence of VPCI. The surface of mild steel coupon treated with CA and BA are smooth and clear which confirms the high PCIE shown by CA and BA against the atmospheric corrosion. There is uniform type of corrosion on mild steel coupons treated with DMPU.

IV. MECHANISMS OF INHIBITION

Inhibition of metallic corrosion in presence of investigated VPCIs involves the vaporization of the VPCIs in non dissociated molecular form and followed by the adsorption of the vapors of VPCIs on the metal surface due to the presence of lone pairs of electrons on hetero atoms of inhibitors. VPCI functions by forming a bond on the metal surface and by forming a barrier layer to aggressive ions. On contact with the metal surface, the vapors of the VPCI are condensed and are hydrolyzed by moisture to release protective ions. A VPCI must be capable of forming a protecting layer that limits penetration of the corroding species [28, 29].

The VPCIs investigated in present study inhibited corrosion of metals in various ways:

- By saturating the space with their vapors and reducing the relative humidity below critical value.
- By alkalizing the medium to a pH value at which the rate of corrosion become significantly low.
- By reducing the corrosion current density to a minimum value by rendering the metal surface hydrophobic which prevented the reaction of metal with environment.
- The presence of more number of lone pairs in the inhibitor enhances their inhibition efficiency

but presence of unsaturation and bulky alkyl groups near lone pair carrier atom retard their action due the resonance stabilization and steric hindrance respectively.

Very high percentage corrosion inhibition efficiency shown by CA and BA may be due to the following reasons:

- 1) By saturating the space with their vapors (due to high vapor pressure) and reducing the relative humidity below critical value.
- 2) Due to presence of lone pair on oxygen atom and presence of saturated aliphatic chains leads to stronger adsorption to metal which forms uniform protective barrier film over the surface of metal.
- 3) Low percentage corrosion inhibition efficiency shown by DMPU and ABT may be due to their low vapor pressure and resonance stabilization of lone pair of electrons on nitrogen and sulphur atom in ABT and two methyl group on nitrogen cause steric hindrance in DMPU.

V. CONCLUSION

From the results of Weight Loss, Salt Spray, Eschke test and SEM techniques, the following conclusion can be drawn.

- 1) All the four investigated VPCIs show high percentage corrosion inhibition efficiency toward mild steel in different corrosive environment like high relative humidity, 3.0 % sodium chloride and high temperature i.e. 40.0 °C.
- 2) Out of four investigated VPCIs, n-Caprylic acid (CA) and n-Butyric acid (BA) shows high corrosion inhibition efficiency in different corrosive environment.
- 3) VPCI saturate the space with their vapors and reducing the relative humidity below critical value and also alkalize the medium to a higher pH value at which the rate of corrosion become significantly low.
- 4) Percentage corrosion inhibition efficiency was found to be in the order n-Caprylic acid > n-Butyric acid > 2-Amino Benzothiazole > N,N-dimethyl propylene urea in weight loss and salt spray corrosion experiments.
- 5) It was observed that aromatic nitrogen bases like ABT shows slightly less percentage corrosion inhibition efficiency (80.0 %) whereas aliphatic compounds like n-Caprylic acid (CA) and n-Butyric acid (BA) shows high PCIE (99.0 %) for mild steel.
- 6) Results obtained from Weight Loss technique, Eschke test, Salt Spray method are in good agreement with each other inspite of different corrosive environment and are further supported by surface study carried out by SEM technique.

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