Corrosion behavior of metals and alloys in marine-industrial environment

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Abstract
This work deals with atmospheric corrosion to assess the degrading effects of air pollutants on ferrous and non-ferrous metals and alloys, which are mostly used as engineering materials. An exposure study was conducted in the Tuticorin port area located on the east coast of South India, in the Gulf of Mannar with Sri Lanka to the southeast. Common engineering materials, namely mild steel, galvanized iron, Zn, Al, Cu and Cu–Zn alloys (Cu–27Zn, Cu–30Zn and Cu–37Zn), were used in the investigation. The site was chosen where the metals are exposed to marine and industrial atmospheres. Seasonal 1 to 12 month corrosion losses of these metals and alloys were determined by a weight loss method. The weight losses showed strong corrosion of mild steel, galvanized iron, Cu and Zn and minor effect on Al and Cu–Zn alloys. Linear regression analysis was conducted to study the mechanism of corrosion. The composition of corrosion products formed on the metal surfaces was identified by x-ray diffraction and Fourier transform infrared spectroscopy.

Keywords: atmospheric corrosion, ferrous, nonferrous metals and alloys, marine-industrial areas, regression analysis, corrosion products analysis

(Some figures in this article are in colour only in the electronic version)

1. Introduction
Corrosion behavior of metals and alloys is mainly affected by their environment. In open atmosphere, corrosion depends on seasonal weather variations. Atmospheric environments can be classified as marine, industrial, urban and rural.

Many investigators have examined the corrosion rates of various metals exposed to different atmospheres [1–6]. Exposure studies were conducted to evaluate the relative corrosion resistances of various metals to different atmospheric environmental conditions. It is well accepted that the cost of material deterioration in an atmospheric environment is enormous, and that almost one-half of this cost is due to the corrosion of steel [7]. Therefore, galvanized protective coatings are essential for the economic use of steel [8]. Copper and its alloys have wide applications both contemporary and historic. These are the main materials used in electrical power lines because of their high conductivity. These materials also have several applications in the electronic and electrochemical industries. In addition, copper and its alloys have good resistance against atmospheric corrosion [9] and are resistant to bio-fouling [10]. Corrosion losses of Fe, Cu, Zn [11] and Al were found to depend on relative humidity, wetting time, amount of chloride and presence of SO\textsubscript{2} in the atmosphere. High corrosion rates in industrialized areas have therefore been related to the high SO\textsubscript{2} concentrations present in these areas. The chloride content in the marine area has also been shown to substantially affect the corrosion of Fe [12, 13], Zn [14], Cu [15] and Al [16]. The high resistances of Zn and Cu against atmospheric corrosion compared with Fe were attributed to the formation of basic corrosion products [17].
in which aggressive ions are captured and inactivated. Good corrosion resistance of Al was explained by amorphous hydroxides immobilizing aggressive ions [18]. Atmospheric corrosion phenomena have been widely studied and analyzed by statistical models based on regression analysis [2,19–22]. Lien et al. [23] studied the corrosion behavior of carbon steel, Zn and galvanized steel at 6–8 exposure sites in Vietnam over a period of ten years. Their results showed that the corrosion mainly depend on the wetting time and amount of chloride in the atmosphere. Dong et al. [24] introduced the atmospheric corrosion studies and climatic classification of the atmospheric corrosion in China.

There are many studies on environmental corrosion over a large variety of geographic zones with widely varying environmental and meteorological conditions [25–29]. However, the number of studies, which focus on environmental corrosion in marine-industrial areas, is fairly limited [30–32].

The first corrosion map of India was developed 36 years ago [33]. Many environmental changes have occurred due to industrialization, population growth and climatic changes. A new corrosion map of India is being prepared by the Central Electrochemical Research Institute, Karaikudi, India, and some data have already been published [34–39].

Tuticorin (marine-industrial site) is one of the 41 field exposure sites covering the whole India. This site is located on the Bay of Bengal seacoast surrounded by many chemical and fertilizer plants, a thermal power station and the port of Tuticorin, and it is 5 km away from the city. Exposure stands were erected within the campus of Tuticorin port area. The port is situated at latitude 8°45′ north and longitude 78°13′ east on the east coast of India. It is located in the Gulf of Mannar with Sri Lanka to the southeast.

In this paper, we present the data obtained from the seasonal 1 to 12 month atmospheric exposures of mild steel, galvanized iron, Zn, Al, Cu–27Zn, Cu–30Zn and Cu–37Zn specimens at the site. We also present the statistical analysis of weight loss data and the characterization of the corrosion products formed on the metal surfaces.

2. Experimental procedure

Atmospheric exposure tests were conducted at the Tuticorin exposure site in India. This site was classified as a marine-industrial site. It is a highly industrial area, which is located near the seacoast, and hence, it is a combination of both marine and industrial environments. Commercial mild steel, Zn, galvanized iron, Al, Cu–27Zn, Cu–30Zn and Cu–37Zn were used in this study. Specimens with dimensions of 100 × 150 mm² were cut from 1-mm-thick sheets and cleaned to remove the scale and other products by pickling. They were polished, degreased, and weighed before exposure. Then, they were positioned on the exposure stands at an angle of 45° facing towards the seacoast under outdoor conditions. The tests were carried out during the period 2000–2001. The metal specimens were removed from the test site after exposure for 1, 3, 6, 9 and 12 month. Then, they were cleaned in respective pickling solutions as described in the ASTM norms G1-90 [40], dried and weighed again. The corrosion losses of the specimens were determined by the gravimetric method in accordance with ISO 9226 [41], and the rate of corrosion loss was obtained as the average of three specimens for each exposure period.

The climatic characteristics of temperature, relative humidity and rainfall were obtained experimentally from the test site. The pollution caused by SO₂ and the airborne salinity represented by chloride were evaluated continuously every month by the deposition methods with a lead peroxide candle and by the wet candle methods, respectively, in accordance with ISO 9225 [42].

The composition of the corrosion products of the exposed samples was determined by x-ray diffraction (XRD, JEOL-JDX-8030) and Fourier transform infrared spectroscopy (FTIR, Perkin Elmer 783).

3. Results and discussion

3.1. Seasonal variations of corrosion rate

Average monthly variations in temperature, humidity and the deposition rates of pollutants, such as chloride and SO₂, are given in table 1. The average annual temperature in this area is around 28–34°C, average relative humidity varies from 70 to 97%, and the annual rainfall is 1–3 mm. The southwestern monsoon period falls between June and July, and the northeastern monsoon occurs between October and November in the coastal belts in Tamil Nadu, India. During

| Month, year | Max. Temp (°C) | Max. Rainfall (mm) | Max. RH (%) | SO₂ (mg (m²d)⁻¹) | Cl⁻ (mg (m²d)⁻¹) |
|------------|----------------|-------------------|-------------|-------------------|-------------------|
| Aug. 2000  | 33.1           | 5.1               | 97          | 30.3              | 52.1              |
| Sep.       | 32.1           | 7.7               | 95          | 53.4              | 54.9              |
| Oct.       | 32.8           | 9.6               | 67          | 36.6              | 45.1              |
| Nov.       | 30.3           | 4.9               | 90          | 34.3              | 40.1              |
| Dec.       | 28.7           | 6.7               | 88          | 30.1              | 46.6              |
| Jan. 2001  | 29.0           | 0.58              | 88          | 27.2              | 35.4              |
| Feb.       | 29.8           | 12.6              | 65          | 30.3              | 44.1              |
| Mar.       | 31.9           | 1.9               | 97          | 28.9              | 50.1              |
| Apr.       | 33.4           | 3.1               | 65          | 20.8              | 59.2              |
| May        | 32.5           | 2.1               | 88          | 28.8              | 55.7              |
| June       | 35.5           | 2.4               | 70          | 22.9              | 50.2              |
| July       | 34.7           | 1.8               | 85          | 27.4              | 38.0              |

| Month, year | Max. Temp (°C) | Max. Rainfall (mm) | Max. RH (%) | SO₂ (mg (m²d)⁻¹) | Cl⁻ (mg (m²d)⁻¹) |
|------------|----------------|-------------------|-------------|-------------------|-------------------|
| March 2001 | 31.9           | 1.9               | 97          | 28.9              | 50.1              |
| April      | 33.4           | 3.1               | 65          | 20.8              | 59.2              |
| May        | 32.5           | 2.1               | 88          | 28.8              | 55.7              |
| June       | 35.5           | 2.4               | 70          | 22.9              | 50.2              |
| July       | 34.7           | 1.8               | 85          | 27.4              | 38.0              |

Table 1. Climatic and pollutant data for the Tuticorin marine-industrial site. RH is relative humidity.
the night, the temperature drops to the dew point and thus thin films of moisture are formed on the surfaces. Those films evaporate during the day. The deposition rates of chloride and SO$_2$ range from 35 to 59 mg (m$^2$d$^{-1}$) and from 208 to 234 mg (m$^2$d$^{-1}$), respectively. Small variations indicate that the deposition rates of these pollutants are almost constant throughout the exposure period. High humidity and pollution levels are the main corrosion factors of the atmosphere. In accordance with ISO 9223 [43], pollution at the Tuticorin test site caused by SO$_2$ was classified as P$_3$ (the highest possible category), chloride pollution was classified as S$_1$ (medium), and the site was designated as an S$_1$P$_3$ mixed environment.

Seasonal variations in the weight losses of mild steel, galvanized iron, Zn, Al, Cu and three Co–Zn alloys are shown in figures 1 and 2 as a function of exposure period. The weight loss of mild steel varies from 3.75 to 12.79 mg cm$^{-2}$ depending on exposure period; the weight loss was higher in November and lower in January. For galvanized iron and Zn, the weight losses were 0.51–3.5 mg cm$^{-2}$ and 0.89–3.37 mg cm$^{-2}$, respectively; these values are 2 to 5 times smaller compared to mild steel. The weight loss was high in September and October for galvanized iron and in November for Zn; it was low in July for galvanized iron and in June for Zn. Those variations light originate from the wet and dry seasons of the exposure site. For Al, high weight loss in October and November due to the high humidity and pollution levels is revealed in figure 1. The high loss of Al in November indicates that corrosion is dominated by pre-existing defects (e.g. pores and pinholes) in the aluminum oxide coating of the metal surface. Airborne chloride ions diffuse into the oxide layer via ionic vacancies, forming soluble complex hydroxide chloride, which locally dissolves the oxide layer. This leads to local acidification in the pit with HCl formation, which accelerates Al corrosion.

3.2. Kinetics of corrosion process

The weight loss variations after 1, 3, 6, 9 and 12 month exposures of mild steel, galvanized iron, Zn, Al, Cu and Cu–Zn alloys are shown in figures 3 and 4. These plots represent the average weight loss obtained from three specimens. The variations observed during the exposure can be classified into two classes corresponding to the difference in corrosion behavior. Corrosion of Zn and Cu appears similar as the weight loss curves exhibit parabolic time dependence in the initial period of exposure, followed by a continuous decrease after exposure for few months. The increase in weight loss observed in the first few months is due to the non-uniform coverage of the corrosion layer over the surface. The weight loss recorded after that depends on the rate of continuous removal of corrosion products with dew and rain. In the initial stage, the formation rate of corrosion product is higher than the dissolution rate of the corrosion layers. After certain time of exposure, the corrosion layers reach a critical thickness, and we assume that the diffusion process...
observed in the layers, and therefore the corrosion of the substrate, slow down [44]. This slowing down is probably due to the formation of adherent corrosion products on the surface. The film of corrosion product becomes denser with continued exposure, thus affording a thicker, more protective coating [45]. Visual observation of exposed Zn and Cu specimens reveals that Zn changes from bright lustrous zinc to white rust, and it completely turns into rust after 12-month exposure. In case of Cu, green patinas were formed and distributed non-uniformly over the surface.

The corrosion behavior of mild steel, galvanized iron, Al and Cu–Zn alloys (Cu–27Zn, Cu–30Zn and Cu–37Zn) was linear (figures 3 and 4) throughout the exposure period, in agreement with earlier studies [46–48]. The corrosion products formed on the surface are mostly soluble compounds; hence, a linear variation is observed.

In case of Cu–27Zn, Cu–30Zn and Cu–37Zn alloys, the weight loss increases with exposure period owing to a dezincification process [49–51], which is directly related to the Zn content in the Cu–Zn alloys. It can be seen in figure 4 that the weight loss of the Cu–37Zn alloy with a higher Zn content is higher than in Cu–27Zn and Cu–30Zn. These Cu–Zn alloys can be ranked in the order of decreasing atmospheric corrosion resistance as Cu–37Zn, Cu–30Zn, Cu–27Zn, Cu–37Zn. These Cu–Zn alloys reveal less cuprous oxide in the corrosion product, as well as increased amounts of cupric oxide and chlorides as basic zinc chlorides; there is also strong component of ZnO formed as a result of dezincification.

The results of the present and previous studies [56–59] suggest that galvanized iron, Al, Cu and Cu–Zn alloys have lower weight losses than mild steel after 12 month exposure to marine-industrial environment; dezincification leads to a higher weight loss for Cu–37Zn than for Cu–Zn alloys with lower Zn fraction.

After re-plotting weight loss data in logarithmic scales, a linear relationship is observed, which can be expressed as

$$\log C = \log A + B \log t. \quad (1)$$

Here C is the weight loss in mg cm\(^{-2}\), \(t\) is time in months, A and B are constants. Using the above equation, the data were analyzed and the values are given in table 2.

### Table 2. Kinetic parameters: weight loss for first month, constants A and B of power functions \(C = A t^B\), and correlation coefficient \(R^2\). MS is mild steel and GI is galvanized iron.

| Metal       | Actual value (first month, mg cm\(^{-2}\)) | Predicted value (mg cm\(^{-2}\)) | Relative prediction error (%) | B     | \(R^2\) |
|-------------|---------------------------------|---------------------------------|--------------------------------|-------|--------|
| MS          | 9.43                            | 9.28                            | ~1.5                           | 0.54  | 0.98   |
| Zn          | 1.60                            | 2.35                            | –                              | 0.09  | 0.01   |
| Cu          | 1.51                            | 1.9                             | –                              | 0.07  | 0.02   |
| Cu–27Zn     | 0.57                            | 0.57                            | 0                              | 0.41  | 0.98   |
| Cu–30Zn     | 0.67                            | 0.68                            | ~1.0                           | 0.38  | 0.98   |
| Cu–37Zn     | 0.73                            | 0.71                            | ~1.4                           | 0.43  | 0.99   |

All the weight loss data agree fairly well with the plots of \(\log C\) versus \(\log t\), thus proving that our data obey the exponential dependence

$$C = A t^B. \quad (2)$$

Equation (2) is often applied to assess the long-term corrosion of metals from the experimental results obtained in short-term tests [2, 9, 22]. Reasonable agreement is observed between the calculated and observed corrosion losses for short-term tests. The corrosion rates observed at the Tuticorin site and estimated by extrapolating equation (2) are also given in table 2. The predicted relative error is under 11% for all metals.

Table 2 lists B values for Zn and Cu as 0.07 and 0.09, respectively, and for Cu–Zn alloys, they are ~ 0.5 suggesting that corrosion is a diffusion-controlled mechanism [60]. On the other hand, the B values for mild steel, galvanized iron and Al are > 0.53 indicating that corrosion is controlled by the charge transfer mechanism [61].

### 3.3. Characterization of corrosion products

#### 3.3.1 X-ray diffraction analysis

XRD patterns were obtained for 2θ angles between 10° and 70°. The phases present were identified using the 1997 PCPDFWIN program. Figures 5–7 show the XRD patterns of Cu, Zn and Cu–37Zn exposed at the Tuticorin site. The dominant phase in the patinas is cuprite (Cu\(_2\)O, figure 5), and basic copper chlorides are detected in the form of atacamite Cu\(_2\)(OH)\(_3\)Cl, posnjakite Cu\(_4\)SO\(_4\)(OH)\(_6\)·2H\(_2\)O and brochantite Cu\(_4\)SO\(_4\)(OH)\(_6\). The XRD patterns for Zn (figure 6) reveal simonkolleite Zn\(_6\)(OH)\(_8\)Cl\(_2\)·2H\(_2\)O and zinc carbonate hydroxide Zn\(_4\)Cl\(_2\)(OH)\(_4\)SO\(_4\)·5H\(_2\)O. The marine-industrial atmosphere is strong oxidizing agent as revealed in figure 6 by small height of the Zn peak.

For the Cu–37Zn alloy (figure 7), the XRD pattern show less cuprous oxide in the corrosion product, as well as increased amounts of cupric oxide and chlorides as basic zinc chlorides; there is also strong component of ZnO formed as a result of dezincification.
Typical diffraction patterns of corrosion products formed on mild steel and Al are shown in figures 8 and 9, respectively. The corrosion products of mild steel are predominantly lepidocrocite ($\gamma$-FeOOH) and goethite ($\alpha$-FeOOH). Soluble compounds FeSO$_4$ and FeCl$_3$ were also identified.

The corrosion of the Al layer is strongly affected by airborne chloride followed by SO$_2$. The XRD results (figure 9) show that the corrosion products consist of Al$_3$(SO$_4$)$_2$(OH)$_4$·9H$_2$O and aluminum oxide or hydroxide. The corrosion layer flaked off the base metal owing to an increase in volume during its transformation from aluminum oxide or hydroxide to the above-mentioned sulfate.

3.3.2 FTIR spectroscopy: FTIR spectra were recorded in transmittance mode at a resolution of 4 cm$^{-1}$ and interpreted using the previous data [62, 63]. Figure 10 shows the spectra of mild steel, Al, Zn, Cu and Cu–37Zn. The corrosion products formed after 12-month exposure were investigated in the range 500–4000 cm$^{-1}$. The spectrum of Cu corrosion products shows (curve d in figure 10) strong bands at around 3441 and 3450 cm$^{-1}$ assigned to the stretching vibrations of hydroxyl groups of atacamite [64] and water. Weak band at 1632 cm$^{-1}$ is OH deformation vibration. The band characteristic of cupric sulfate [65] is also observed around 1110 cm$^{-1}$. The absorption band at 625 cm$^{-1}$ [66] is assigned to cuprite (Cu$_2$O). The spectra also show peaks below 500 cm$^{-1}$ characteristic of Cu–O bonds.

The spectra of Zn corrosion products in the range 350–600 cm$^{-1}$ (curve c in figure 10) correspond to Zn–O bond and reveal that ZnO (zincite) is formed. Zinc oxide formed under dry condition undergoes further transformation to Zn(OH)$_2$ in presence of water. If chloride is present in the atmosphere then zinc hydroxide reacts with chloride to form insoluble simonkolleite:

$$\text{ZnO} + 2\text{Cl}^- + 6\text{H}_2\text{O} \rightarrow \text{Zn(OH)}_6\text{Cl}_2 \cdot 2\text{H}_2\text{O} + 2\text{(OH}^-). \quad (3)$$

The spectrum indicates that major corrosion products for Zn during wet and dry seasons are zinc hydroxy carbonate, hydroxy chloride and hydroxy sulfate compounds possibly due to their high solubility.

The spectra of corrosion products of Cu–37Zn brass are dissimilar to those of Cu and Zn with the appearance of several additional weak bands (curve e in figure 10).
The FTIR spectra of Cu–37Zn show major changes in the range 450–1600 cm\(^{-1}\). Stretching and deformation modes of OH group are observed between 3530 and 2500 cm\(^{-1}\) and at 1637 cm\(^{-1}\), respectively. Copper sulfate, CuO and ZnO are detected via the absorption bands at 1121 cm\(^{-1}\), 610 cm\(^{-1}\) and 450 cm\(^{-1}\), respectively. These absorption bands suggest presence of CuCO\(_3\), Cu(OH)\(_2\), ZnO, CuO and CuSO\(_4\) phases, which were also detected by XRD. Absence of the characteristic Cu\(_2\)O peak at 625 cm\(^{-1}\) indicates that the corrosion products formed on the Cu–37Zn surface mainly consist of Zn compounds due to the dezincification of brass under atmospheric exposure.

Curve (a) of figure 10 shows the FTIR spectra of the corrosion products formed on mild steel exposed for 12 months. The absorption peaks at 1110 and 1020 cm\(^{-1}\) and weak peaks at 740 and 490 cm\(^{-1}\) reveal presence of \(\gamma\)-FeOOH. Both \(\alpha\) and \(\delta\) phases exhibit two characteristic absorption peaks at 885 and 798 cm\(^{-1}\) and overlap, whereas the \(\delta\) phase shows a distinct and broad peak at 1110 cm\(^{-1}\). The FTIR spectrum clearly confirms that the \(\gamma\)-phase does not occur separately.

Absorptions at 546, 1630 and 3462 cm\(^{-1}\) reveal free surface hydroxy groups on Al (curve b in figure 10).

4. Conclusions

Ferrous and non-ferrous metals and alloys exposed to a marine-industrial atmosphere site exhibit a surprisingly large difference in corrosion behavior. The seasonal variations in the corrosion rates of these metals depend mainly on the atmosphere chloride, SO\(_2\), and humidity. The exposure site was classified as S\(_1\)P\(_3\) corrosive environment in accordance with ISO 9223. Mild steel, galvanized iron, Al and Cu–Zn alloys after 1, 3, 6, 9 and 12 month exposures showed a linear dependence of weight loss, whereas Cu and Zn exhibited a parabolic time dependence of weight loss with exposure time. Regression analysis suggests diffusion-controlled corrosion for Zn, Cu and Cu–Zn alloys, whereas charge-transfer-controlled process can be assigned to other metals. Corrosion of Cu–27Zn, Cu30Zn and Cu–37Zn mainly depends on Zn content in the alloys, and these alloys, were ranked in the order of decreasing atmospheric corrosion resistance as Cu–37Zn, Cu–30Zn and Cu–27Zn. The main corrosion products formed on the metals were identified by XRD and FTIR techniques.

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