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Corrosion Behaviour of Mild Steel in Atmospheric Exposure and Immersion tests

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ABSTRACT: Corrosion testing is an essential tool in the development of corrosion protection strategies. Its prominence cannot be overlooked in this world of limited resources. Accelerated tests including the alternate immersion test and the simple immersion test, were performed according to ISO 11130 and ASTM G31 to study the corrosion behaviour of mild steel. A time correlation has been established between these tests and the corrosion loss in a specific atmosphere. They were also compared with atmospheric exposures at a marine site in Mauritius. Test specimens were visually inspected to understand the corrosion behaviour in these accelerated tests. The study concluded that a concentration other than 3.5% NaCl should be envisaged for the alternate immersion test to at least follow a trend similar to that of atmospheric corrosion.

KEYWORDS: corrosion, immersion tests, atmospheric, mild steel, NaCl

I. INTRODUCTION

Corrosion tests can be used to monitor the corrosion behaviour of materials in specific environments. They are classified into three main groups; exposure tests, pilot tests and laboratory tests. An exposure test involves the exposure of the material to the service environment. Exposure tests provide accurate results but they usually require a few years to provide results. Pilot tests are conducted to compare the corrosion performance of materials in service. They require the substitution of existing components in a system by components made of the material to be assessed. In laboratory tests, the metal is exposed to a more aggressive environment than the actual service environment [1]. In the lifetime prediction of the corrosion performance of materials in specific atmospheres, atmospheric exposures have been very reliable and have been performed worldwide [2]. However, in many circumstances rapid and reliable prediction of the materials' performance in corrosive environments is required and, in these situations accelerated corrosion tests are preferred [3].

The salt spray test is a commonly used accelerated corrosion test [2]. Atmospheric corrosion is highly dependent on pollutants and humidity cycles. The salt spray test, however, involves neither humidity cycling nor SO_2 and thus does not provide the conditions of a general atmosphere. It has been highlighted that the salt fog test induces consistent conditions throughout the test cycle and consequently forces a consistent corrosion rate which does not occur in a real-time atmosphere. Consequently, the results obtained do not correlate satisfactorily with those from outdoor exposures for a general use [2].

II. RELATED WORK

Immersion tests can be used as an inexpensive alternative to salt spray tests. They involve the immersion of the specimens in a corrosive liquid and measurement of mass loss for a period of time. Two common types of immersion tests are:

• The Simple Immersion test

It involves the complete immersion of the specimens in a corrosive liquid and measurement of mass loss for a period of time. It is the most economical method for assessment of materials and eliminating those materials that must not be



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used for specific applications. However, the lifetime of a system cannot be predicted by extrapolating results from this simple test [8].

• Alternate immersion test

This test involves the repetitive immersion and drying cycles for a specific time period depending on the proneness to corrosion of the specimens and the purpose of the test. It can be performed according to ISO 11130[4] or ASTM G44 [5]. This accelerated corrosion test is able to replicate the corrosive conditions of marine environment, marine splash zones, de-icing fluids and acid salt environments [4].

The present study has been performed to determine the corrosion behaviour of mild steel in alternate immersion, simple immersion tests and atmospheric exposures in order to:

- Understand the difference between alternate immersion and simple immersion tests.
- Obtain a correlation between the accelerated tests and atmospheric exposure. For the alternate immersion test, the number of cycles that need to be performed to achieve the corrosion losses equivalent to the different corrosion categories of ISO 9223 would be determined, while for the simple immersion test, the number of days of simple immersion required would be determined.
- Compare the corrosion trend of the alternate immersion test to that of the atmospheric exposure at a marine site in Mauritius, namely Belle Mare.

It should be noted that results of alternate immersion tests have been rarely published and hence this paper aims at giving an insight to the corrosion behaviour of mild steel in this test and also the comparison of the corrosion behaviours in this study will prove to be very useful to individuals related to this field.

The location of Mauritius in the Indian Ocean is shown in Fig.1. The marine site of Belle Mare is found on the eastern coast of Mauritius. Mild steel samples were exposed 50m from the sea shore and the corrosion loss (in μ m) was determined through the mass loss method [6].



Figure 1: Location of Mauritius

The corrosion loss was plotted against the time of exposure on logarithmic scales as shown in Fig. 2. It was observed that the corrosion loss at Belle Mare is given by Equation 1:

$$C = t^{0.6}$$
 Equation 1

where C is the corrosion loss (μm) and t is the time the of exposure (in terms of days)



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Figure 2: Corrosion loss resulted from atmospheric exposures at Belle-Mare

The level of airborne salinity and sulphur dioxide level were also monitored for the exposure period. The average levels, according to ISO 9223[6], during this time period are shown in Table 1[7].

	U
Chloride class	\mathbf{S}_1
SO ₂ class	Po
Time of wetness class	T_4
Corrosivity category based on environmental parameters	C3

Table 1: Airborne salinity and sulphur dioxide level according to ISO 9223[7]

Using Equation 1, it was found that the corrosion rate at Belle Mare falls in the corrosivity category C3, according to ISO 9223, which is same as that obtained in Table 1. The results of the atmospheric test at Belle Mare would be essential for comparison with those of the alternate immersion test. The latter could then be used for predicting atmospheric corrosion in the marine atmosphere of Mauritius.

III. METHODOLOGY

Mild steel samples of size $120\text{mm} \times 90\text{mm} \times 0.68\text{mm}$ were used for the alternate immersion test while samples of size $100\text{mm} \times 60\text{mm} \times 0.76\text{mm}$ were used for the simple immersion test. They were manufactured according to SABS 1431. After cutting the samples to size, they were cleaned in dilute acid, followed by neutralization of the acid and then passivation of the surface. The test solution is another essential component of the alternate immersion test, which consisted of 35g.L-1 sodium chloride solution and was prepared according to ISO 11130[4].

The alternate immersion test was carried according to ISO 11130 and ASTM G44[5]. It was performed in a clean room at a temperature of 26°C and relative humidity of 50% with an equipment made according to these two standard guides. The corroded specimens were removed in batches of 3 after 16, 34, 58, 86, 102 and 126 cycles. Upon removal the samples were cleaned and their average mass loss and, subsequently, corrosion loss (μ m) were determined.

The simple immersion test was performed according to the ASTM G31-12a [8]. It was carried under same room conditions as the alternate immersion test. The samples were completely immersed in 3.5% NaCl solution. They were removed after 2, 6, 8, 10, 12, 14 and 17 days of immersion. The corrosion products, for both the alternate immersion and simple immersion tests were removed according to the ASTM G1-03[9]. Prior to the chemical cleaning, the corroded specimens were cleaned using a soft non-metallic bristle brush and then rinsed using distilled water. Chemical cleaning was carried in 200g of sodium hydroxide pellets and 20g of zinc chips in 1 L of water heated to 80°C - 90°C



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for about 40 minutes. After the removal of the corrosion products, the coupons were rinsed using distilled water and then weighed to determine the mass loss. The corrosion loss was eventually calculated.

IV. RESULTS AND DISCUSSION

The specimen withdrawn after the 5th cycle of alternate immersion is shown in Fig. 3. It was observed that the corrosion attack was highly localised.



Figure 3: Typical sample after 5 cycles of alternate immersion

Fig.4 shows the localised attack on a specimen withdrawn after the 16th cycle of alternate immersion.



Figure 4: Typical sample after 16 cycles of alternate immersion

Initially the samples encountered highly localized attack, which then proceeded as uniform corrosion. Blistering was observed on the surfaces of the coupons as from the 86th cycle. Alongside blistering, flaking of the corrosion products was also observed at the 126th cycle (Fig.5). The three specimens removed after the 126th cycle were found to be perforated (3 to 4 holes of diameter less than 0.5 mm) after the removal of the corrosion products. Blistering and flaking of the edges are common phenomena observed in coupons exposed to the natural environment [10].



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Figure 5: Typical sample after 126 cycles of alternate immersion

A weakly adherent oxide layer started to appear on the samples after two days of simple immersion. Figure 6 shows the oxide layer on a typical coupon's surface after two days. There was no significant disparity on the appearance of the oxide layer on the coupons removed after 17 days of immersion. It remained weakly adherent.



Figure 6: Weakly adherent oxide layer on a typical sample after 2 days of simple immersion

Figure 7 illustrates the results obtained by performing weight loss analysis of the samples. The equation of the trend line is:

$$= 7.0e^{0.02t}$$

С Equation 2 where C is the average thickness loss in µm and t is the number of cycles over which the samples were tested. The coefficient of determination was found to be equal to 0.99, which is a high value indicating good linear correlation



Figure 7: Results of the alternate immersion test obtained through weight loss analysis

between C and t.



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For atmospheric exposures, the equation for corrosion loss is of the type [10,11]:

$$C = At^B$$

Equation 3

where C is the corrosion loss and t is the time of exposure. A is generally considered as a measure of the initial reactivity of the material with the environment and it is numerically equivalent to the corrosion loss when time is unity. The exponent B is in turn a function of the type of atmosphere where the metal is exposed. In some studies, t has been taken as the time of wetness in the period under consideration [12].

It is well known that the corrosion of steel in the atmosphere follows a mechanism controlled by the diffusion of reactants across the rust layer [13]. The value of B, in Equation 3 usually lies between 0.4 and 0.8 [14]. A value close to 0.5, results from an ideal diffusion controlled mechanism when all the corrosion products remain on the surface. This situation seems to occur in slightly polluted inland atmospheres [15]. The value of B is greater than 0.5 when there is an acceleration in the diffusion process, for example, due to cracking or flaking of the rust layer. The value of B smaller than 0.5 would result from a decrease in the diffusion coefficient with time through compaction of the rust layer, recrystallization, etc.

Equation 2 shows that the alternate immersion test does not simulate atmospheric corrosion conditions. The exponential function shows that the alternate immersion test accelerates the corrosion process to very high rates which would not be achieved even in the harshest environments.

The results of the simple immersion test are illustrated in Fig. 8. It can be seen that there exists a linear relationship between the corrosion loss and the number of days of immersion.



Figure 8: Results of the simple immersion test

This indicates that the weakly adherent black oxide layer formed on the mild steel surface, when the metal was immersed in the NaCl solution, did not provide sufficient protection to the metal. Moreover, the concentration of the solution was not expected to change significantly, thus leading to a constant corrosion rate According to Revie and Uhlig [16], the FeCl₂ formed at the anodes and the NaOH formed at the cathodes diffuse in the NaCl solution and react to produce Fe $(OH)_2$ away from the metal surface, and any Fe $(OH)_2$ formed on the surface does not protect the metal. The graphs of average mass loss per unit surface area against time in hours for both alternate immersion and simple immersion tests are shown in Fig. 9. It can be depicted that the alternate immersion test provides much more aggressive conditions that the simple immersion tests.



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Figure 9: Graph of average mass per unit surface area loss against number of hours

Both tests were performed under the same surrounding conditions and, therefore, it can be deduced that the alternate wet and dry conditions of the alternate immersion tests were responsible for the higher corrosion rate. The drying process of the alternate immersion test involved evaporation of water from the surface of the coupons, leaving behind a higher concentration of NaCl on the surface which aggravates the corrosive attack [16].Simple immersion tests are mostly used for comparing corrosion rates of different materials in particular corrosion media.

The number of cycles of the alternate immersion test corresponding to the range of the thickness loss of each corrosivity category of ISO 9223, was determined. The results are shown in Table 2. The thickness loss was considered for 1 year exposures.

Corrosivity category	Thickness loss (µm) range for 1 year	Range of number of cycles
C1	y ≤ 1.3	Not sensitive enough for this range
C2	$1.3 < y \le 25$	$0 < x \le 64$
C3	$25 < y \le 50$	$65 \le x \le 98$
C4	$50 < y \le 80$	$99 \le x \le 121$

Table 2:No. of alternate cycles and the corresponding corrosivity categories

The corrosion loss of the C3 category can be obtained by carrying 65 to 98 alternate cycles. It should be noted that the trend curve does not give valid results for the C1 category. For the simple immersion test, it has been observed that the number of days of immersion required, to yield thickness losses equivalent to that of C3, would be greater than 17 (Fig.8).

Fig. 2 shows a typical result for the corrosion behaviour of carbon steel in coastal regions in Mauritius. Comparing the latter with the results obtained from alternate immersion tests, it can be deduced that the alternate immersion test provides harsher conditions than atmospheric exposures in the coastal regions of Mauritius. The trend curves (Equation 1 and Equation 2) are not similar and, hence, the accelerated test does not accurately simulate the atmospheric conditions. Atmospheric corrosion is highly dependent on pollutants and humidity cycles. The alternate immersion test allows humidity cycles but simulates the atmospheric corrosion in an imperfect way as the corrosion products may dissolve in the test solution, which is not the case in real atmospheric corrosion [13]. This gives results which are difficult to correlate. The test solution of the alternate immersion test comprised of a 3.5% NaCl solution at room temperature which provides the maximum relative corrosion rate for iron and steel [16]. Considering this fact, a test solution of NaCl other than 3.5% should be envisaged for the corrosion trend to at least resemble that of atmospheric exposures.



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V. CONCLUSION

The alternate immersion test provides the harshest environment conditions as compared to those of simple immersion test and atmospheric exposures and consequently the rate of corrosion attack is highest for the alternate immersion test. The number of alternate cycles required to obtain the corrosion losses of the C3 category of ISO 9223 was found to be between 65 and 98 while for the immersion test it was observed that more than 17 days will be required to yield this amount of thickness loss. A comparison between real-time atmospheric exposures at Belle-Mare and the alternate immersion test revealed that the corrosion trends are completely different and hence this causes the correlation between atmospheric exposures and alternate immersion tests to be difficult. A test solution of concentration other than 3.5% NaCl should be envisaged for the corrosion trend of the alternate immersion test to at least follow that of atmospheric exposures in a similar way.

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REFERENCES

- D.E.J Talbot and J.D.R. Talbot, "Corrosion science and technology", CRC Press, 2nd ed., 2007. 1.
- E. Montgomery, J. Curran, L. Calle and M. Kolody, "Timescale Correlation between Marine Atmospheric Exposure and Accelerated Corrosion 2. Testing, Part 2", Corrosion Conference & Expo., NACE, 2012.
- 3. A.G. Forshee, "Accelerated Corrosion Testing, Part I: An Overview of 20 Possible Accelerated Corrosion Tests", Metal Finishing, pp. 50-54, 1993.
- ISO, "ISO 11130: Corrosion of metals and alloys-Alternate immersion test in salt solution", 2010. 4.
- 5. NACE,"ASTM G44: Exposure of Metals and Alloys by Alternate Immersion in Neutral 3.5 % Sodium Chloride Solution", 2000.
- ISO, "ISO 9223: Corrosion of metals and alloys- Corrosivity of atmospheres- classification, determination and estimation", 2010. 6.
- B.Y.R. Surnam, "Atmospheric corrosion of carbon steel in Mauritius", University of Mauritius, 2010. 7.
- NACE, "ASTM G31-12a: Standard Practice for Laboratory Immersion Corrosion Testing of Metals", 2012. 8.
- NACE, "ASTM G1-03: Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens", 2003. 9.
- Feng-I Wei, "Atmospheric corrosion of carbon steels and weathering steels in Taiwan", Br. Corros. J., Vol. 26, No. 3, pp.209-214, 1991. M. Morcillo, "Atmospheric corrosion map for steel in Canary Isles", Br. Corros. J, Vol. 36, No. 4, pp. 266-270, 2001 10.
- 11.
- 12. L. Veleva and L. Maldonado, "Classification of atmospheric corrosivity in humid tropical climates", Br. Corros. J., Vol. 33, No. 1, pp. 53-57, 1998
- 13. M. Benarie and F.L. Lipfert, "A general corrosion function in terms of atmospheric pollutant concentrations and rain pH", Atmos. Environ., Vol. 20, No. 10, pp.1947-1958, 1986
- 14. D. Landolt. "Corrosion and surface chemistry of materials", CRC Press, 3rd ed, pp. 352-357, 2007
- M. Morcillo, S. Feliu and J. Simancas, "Deviation from bilogarithmic law for atmospheric corrosion of steel", Br. Corros, J., Vol. 28, No. 1, pp. 15. 50-52, 1993
- 16. W.R. Revie and H.H. Uhlig, "Corrosion and Corrosion Control", John Wiley & Sons, 4th ed., pp. 132, 2008