Accelerated Test Methods for Evaluation of WB Coating System Comprising of Epoxy Primer and Polyurethane Top Coat

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ABSTRACT

In corrosion protection study, it is important to develop economically attractive test method for mitigation of corrosion damage of coated structure. Faster predication of protective behavior of any coating is a big challenge for the coating industries. In present work, in-house waterborne anticorrosive coating system was selected for study. The corrosion protection of coating evaluated as per ISO12944-C3-High which includes salt spray (ISO 9227, 480 h) and quick condensation test (ISO6270, 240 h). The exposure period was extended up to 1000 h for further monitoring of deterioration of selected coating system. In view of early predication of coating performance, different accelerated test exposure conditions were designed in laboratory, such as thermal aging in distilled water (50°C and 80°C), chemical immersion (H_2SO_4 and NaOH), wet-dry cycle and thermal cycling. The corrosion protection properties of coating exposed to ISO 12944-C3-High environment was compared against the lab designed test method using electrochemical impedance spectroscopy (EIS) technique. The deterioration rate of coating was measured by evaluating polarization resistance (Rp), water uptake (w) and break point frequency (F_b). The coated specimens after exposure were also evaluated for loss of adhesion strength. Results showed that thermal aging in distilled water at 80°C is the most severe exposure condition as coating system loses its protective behavior in relatively short period. Prog. Color Colorants Coat. 10 (2017), 93-104© Institute for Color Science and Technology.

1. Introduction

Organic coatings are widely used for protection of metallic structures against corrosion. Due to strict Environment, Health and Safety (EHS) regulation and laws, coating industry is moving towards a solution which can drastically reduce the use of volatile organic compounds (VOCs). Among the various solutions, use of waterborne coating is the most emerging area [1, 2]. These coatings do not contain flammable or health hazardous solvents and have low content of VOC leading to a significantly reduced environmental impact. Waterborne coatings, if properly formulated, can perform similar or even better than its solvent borne counterparts. Many researchers have reported that waterborne coatings can perform exceptionally well in mild to severe corrosive environment [3, 4].

The corrosion resistance of coating is determined by outdoor exposure and various standard accelerated test methods such as salt spray (ISO 9227), cyclic testing (ASTM G85), advanced cyclic testing (ASTM 5894) and quick condensation test (ISO 6270). All the accelerated tests, including outdoor exposure are

relative tests. The standard tests methods are based on visual evaluation which makes it very subjective. These test results provide an indication of relative performance and they heavily depend on the operator to translate the visual observation in to performance rating. The performances of coated panels are also frequently determined by rust creeping from the artificial made scribe. However, electrochemical methods provide results in a short time. The obtained quantified data can give indications on the actual corrosion mechanisms. In addition, corrosion and coating damage may be determined prior to its visual manifestation [5-7].

High performance anticorrosive coating systems behave as a high resistive insulator. The degradation in such coatings can be accelerated by various methods like, thermal aging, UV irradiation and exposure to corrosive environment. Various accelerated test methods were attempted by researchers to accelerate the coatings degradation for faster evaluation of coatings [8-10]. Miszczyk et al. [11] have reported that cyclic thermal shock treatment on coatings from 20°C to 65°C and back to 20°C within three hours reduces the polarization resistance of coating after 6 thermal shocks. S. Brunner et al. [12] have used accelerated UV weatherometer along with sulfuric acid aerosol for life prediction of organic coatings used for exterior application. Gordon Bierwagen et al. have evaluated exterior coatings such as aircraft and pipeline coatings by cyclic exposure to QUV accelerated weatherometer for one week followed by exposure for one week to fog/dry cycle. A lifetime prediction protocol was also proposed by these authors [13]. However, the protocol suggested may not hold true if the cyclic test conditions are different than proposed. A new approach to accelerate the drying and wetting effect on coating has been attempted by Allahar et al. [14] using hydrophilic room temperature ionic liquids.

The present study aims at understanding performance of WB coating in standard accelerated test method (ISO 12944 C3- high) as well as lab designed test conditions. The performance is evaluated under each test conditions to provide an insight about the suitable test methods for early prediction of coating performance using EIS technique.

2 Experimental

2.1. Coatings used for the study

In-house developed high performance coating system which consists of waterborne epoxy primer and waterborne polyurethane as topcoat was selected for the study. The primer was based on solid epoxy dispersion pigmented with steatite, titanium dioxide, modified zinc phosphate and barium sulfate and polyamine dispersion used as curing agent. Topcoat was based on acrylic polyol dispersion cured with hydrophilically modified poly isocyanate. The characteristics of primer and the topcoat are given in Table 1.

2.2. Surface preparation and panel coating

Mild steel panels of size 150 mm x 100 mm x 1.5 mm were used for EIS studies. Before coating application, the surface was cleaned by blasting to Sa 2.5 grade (ISO 8501-1:2007) with chilled iron grit grade no. GC 60 at a pressure of 4.2 kg/cm^2. The blasting profile was 45 - 50 μm and panels were coated immediately after blasting. The application of primer and top coat was carried out using air assisted spray method. After 24 h of priming, top coat was applied. Coated panels were allowed to cure for seven days at ambient condition before testing. The dry film thickness of complete system was 145 ± 15 microns (Primer 75±15 and topcoat 70 ± 15 microns). The edges and back side of panels were sealed with insulated coating before exposure.

2.3. Design of exposure test conditions

The coating degradation was studied using two set of accelerated exposure conditions. First set includes standard exposure conditions followed by evaluation of anti-corrosive properties of the coating, while the second set comprises of lab designed accelerated exposure test conditions. Total six test conditions were designed and their exposure details are given in Table 2 and discussed subsequently. Two set of coated panels were exposed to each test condition to ensure the reproducibility of the results. The coated panels were analyzed by EIS immediately after particular exposure interval for each test conditions.

Table 1: Characteristics of Primer and topcoat.

Properties	Primer	Topcoat
System	Two Pack	Two Pack
Consistency	Thixotropic	Thixotropic
Dry film thickness (DFT)	75 ± 15microns per coat	35 ± 10 microns per coat
% Solid by weight	65	53
Weight/Lit	1.35 ± 0.05	1.20 ± 0.05
Pot life at 30°C	3 Hours	4 Hours

Table 2: Exposure test condition.

Test conditions	
ISO 12944-C3 High	
Test-I	Salt spray (ISO 9227)
Test-II	Quick Condensation test (ISO 6270)
Lab designed test conditions	
Test-III	Hot distilled water immersion test
	(a) Immersion at 50°C
	(b) Immersion at 80°C
Test-IV	Chemical immersion test
	(a) 10 weight % H_2SO_4 solution
	(b) 10 weight % NaOH Solution
Test-V	Wet dry cycle
Test-VI	Thermal cycling

2.3.1. Test-I Salt spray test (ISO 9227)

The test was conducted by continuous exposure of 5 % NaCl solution in salt spray cabinet. The cabinet temperature was about 33.3 - 36.1°C.

2.3.2. Test-II Quick Condensation test (ISO 6270)

This test is a continuous condensation test at constant temperature of 38 ±2°C. The cabinet has been designed in such a manner that coated side of the panels is exposed to a higher temperature (38 ± 2°C) while the opposite side is exposed to ambient temperature. Thus this test induces temperature gradient which can severely damage the coating.

2.3.3. Test-III Hot distilled water immersion

a) Immersion at 50 °C

The glass transition temperature of selected topcoat has a Tg of about 47 °C measured by differential scanning colorimeter (DSC). Most of the organic coatings are formulated to be in a glassy state under normal service conditions in order to obtain characteristics properties.

Above glassy temperature, polymeric chain changes to rubbery state. Due to movement of polymer chains in rubbery state, water molecules can penetrate through the film. In this test, the coated panel was immersed in distilled water at 50°C to understand the performance if exposure temperature is close to glass transition temperature.

b) Immersion at 80°C

As the temperature increases, oxygen concentration decreases and consequently corrosion phenomena decreases. However wicks et al. showed that maximum corrosion of metal occurs at 80°C in distilled water in open environment [15]. Above this temperature, oxygen solubility decreases considerably and hence corrosion reaction subsides. Hence, the coated panels are exposed to distilled water at 80°C for evaluation of the performance of selected coating system under this condition.

2.3.4. Test-IV Chemical immersion test

The coated specimens were immersed in acid and alkali environment i.e. 10 wt% H_2SO_4 solution and 10 wt% NaOH solution, designated as IVa and IVb, respectively.

2.3.5. Test-V Wet dry cycle

Coated panels were immersed in 3.5 wt % NaCl solution at 50°C for 16 h followed by 4 h exposure in cold chamber at 0°C and then 4 h in humidity cabinet, RH 100%. One such a cycle is considered as a complete test. This test simulates the real life environment which is composed of salt fog, temperature change and high humidity. The coating which can sustain its protective behavior may be considered as a good protective coating.

2.3.6. Test-VI Thermal cycling

The test was designed to implement the cumulative effects of thermal cycling [16] on coating-metal substrate interface. The coating was immersed in 3.5 wt% NaCl solution. EIS data were measured from room temperature to 85°C in the following sequence: increasing the temperature from room temperature (25°C) to 45°C, 65°C and 85°C; then decreasing the temperature from 85°C to 65°C, 45°C, and room temperature (25°C). The time period between each temperature change was 1h and the room temperature

immersion was performed for 19 h. The total procedure (5 h temperature variation and 19 h room temperature) was considered as one cycle. This cycle impart the combined effect of varying as well as constant temperature condition on the coating system.

2.4. Evaluation of corrosion resistance using EIS

EIS has been used to study the anticorrosive behavior of anticorrosive coatings [17-19]. Panels removed from each test condition were thoroughly cleaned and then were used for EIS measurement. The impedance measurements were carried out using Gamry CMS 300 electrochemical impedance analyzer. In this study, 3.5% NaCl solution was used as electrolyte and the exposed sample area was 9.6 cm². A three-electrode system having saturated calomel electrode (SCE) as reference electrode, coated metal as working electrode and graphite cylinder as counter electrode was employed. A sine wave of 10 mV (r.m.s) was applied across the cell. The measurements were made in the frequency range of 100 kHz to 0.1 Hz.

2.5. Adhesion strength

Adhesion strength of the coatings was determined by X cut test according to ASTM D 3359. Initial adhesion was checked after 24 h of air drying and wet adhesion was checked after 24 h of completion of each exposure test. Adhesion strength of the coating is measured in A scale in accordance with the specification.

3. Results and discussion

The WB coating system exposed to salt spray (ASTM B 117) and Quick condensation Test (ISO 6270) for 480 h and 240 h, respectively, as per requirement of ISO 12944-C3-high environment. The exposed panels were examined using ISO 4628 standard and the observations are tabulated in Table 3, where deterioration of coating was determined using the scale of 0 to 5. There was no evidence of visible corrosion products after exposure to the particular period. However, further extension of the exposure influences the coating performance. For quantitative test, scribing test panels were used to estimate coating damage and resistance to salt spray exposure (Figure 1). The results reveal that selected WB coating system meets the requirement of ISO12944-C3-high environment.

EIS measurement was performed for WB epoxy-PU

coating system after specified interval for test I to VI. Figure 2 (a) to (f) shows the typical Nyquist plots for WB epoxy coated samples exposed to the various environments. Nyquist plot indicates the relation between imaginary and real impedance for coating and gives information about the protective behavior of the coating. The initial and final periods were selected differently for each test based on the nature of the corrosive environment. The response of impedance magnitude and phase shift varied with exposure duration indicating the decaying nature under a specified corrosive environment. Normally, intact coatings have high resistance. The penetration of water to polymeric film in due course results in the enhancement of the conductivity and switchover from capacitive behavior to a mixed mode i.e. capacitive at high frequency and restive at low frequencies. The decrease in impedance with increasing the exposure time under all test conditions (test I to V) indicates the commencement of corrosion. The Nyquist plots of test IIIB (Figure 2d) and IVB (Figure 2f) reveals the maximum deterioration of coating among all tests.
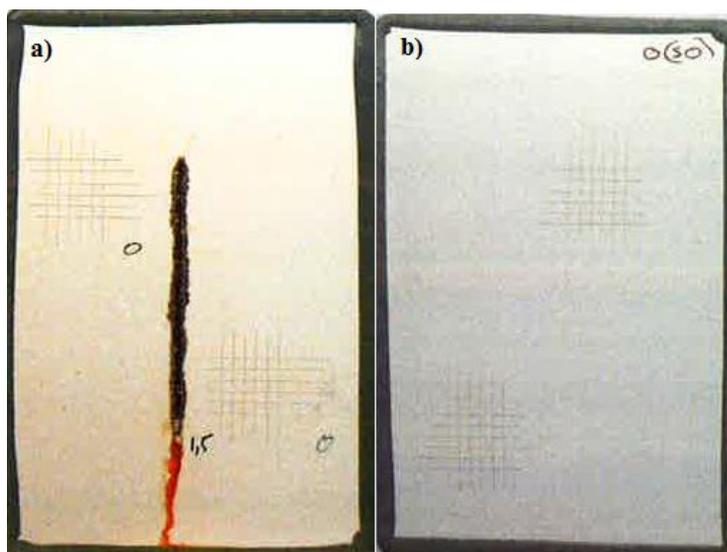
Figure 1: Photographs of test specimens exposed to SS (a) and QCT (b) after 1000 h.
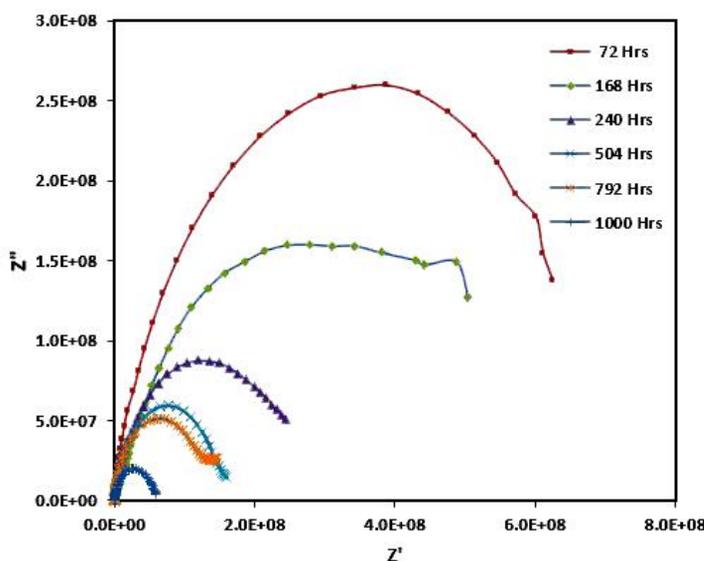
Figure 2: typical Nyquist plots for WB epoxy.

The Bode plot after five cycles and the representative Bode plot after one cycle are shown in Figures 3 (a) and (b), respectively. For understanding the reversible nature of the coating, Bode plot was selected for test-VI. The degree of reversibility of impedance data at low frequency during thermal cycling can be a strong predictor of the corrosion resistance of the samples.

The Nyquist and Bode data of coating exposed to different environments were analyzed with the help of an equivalent circuit model (Figure 4a and b). Figure 4(a) shows the equivalent circuit corresponding to a simple electrochemical corrosion cell. R_s is the solution resistance, R_p is the polarization resistance, and C_{dl} is the double layer capacitance at the metal-solution interface. This model shows a onetime constant impedance spectrum. Figure 4(b) is the equivalent circuit for a coated metal electrolyte system. R_{po} is the pore resistance of the coating and Cc is the coating capacitance. This model gives a two-time constant impedance spectrum. The value of Rs is dependent on the characteristics of the interface between specimen and the electrodes. The change in R_{po} is indicative of the degradation of coated metal systems. By fitting equivalent circuit, different parameters such as polarization resistance (Rp), coating capacitance (Cc) and break point frequency (F_b) are extracted.
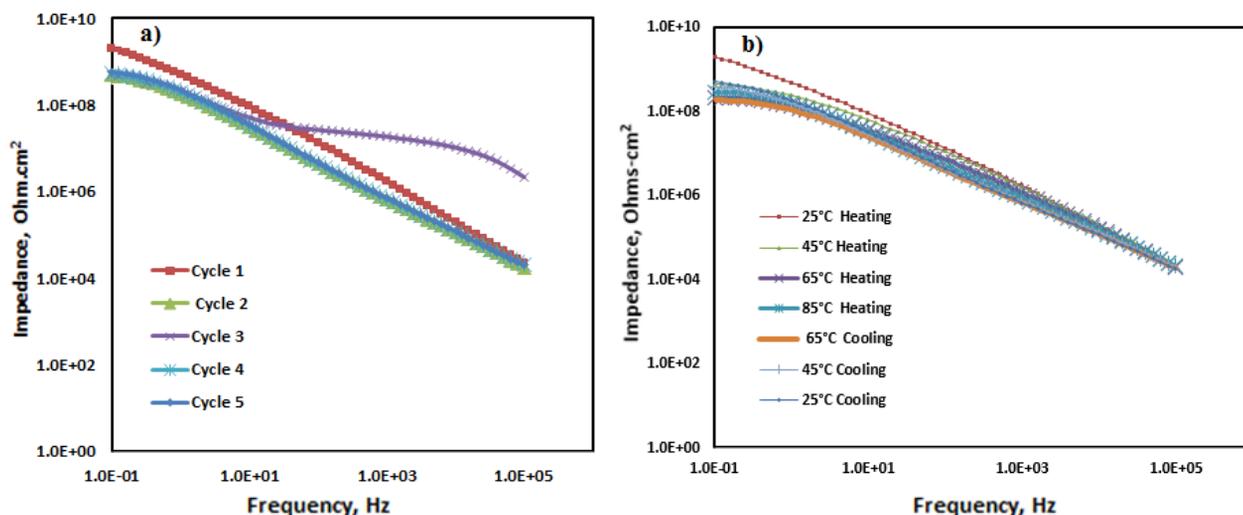
Figure 3: Bode plot of test condition VI: (a) After different cycles and (b) After one cycle.
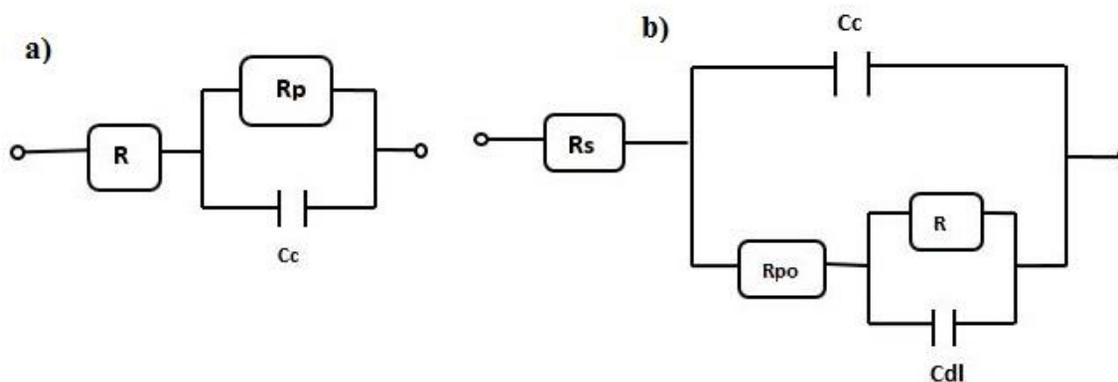
Figure 4: (a) Simple corrosion circuit model (b) Coated steel circuit model.

3.1. Polarization resistance (R_p)

The polarization resistance data of all the coatings was measured using equivalent circuit model. Bierwagen et al. [16, 20-24] used a tentative ranking scheme in their laboratory based on the electrochemical measurement of film resistance in room temperature immersion for intact coatings that gives the probable barrier properties of the coating. The good to excellent coatings polarization resistance values greater than 10^9 Ohm.cm^2 that maintain for >2 weeks in immersion. For fair to good coatings value of 10^6 to 10^9 Ohm.cm^2 that maintain < 2 weeks in immersion. In the case of poor coating R_p values less than 10^6 Ohm.cm^2 that do not maintain even this level of protection in immerse condition. The very good anti-corrosive coating shows higher coating resistance and retains it for a longer time.

It can be seen from Figures 5 (a) and (b) that the polarization resistance decreases with increasing the exposure period for all test conditions (test I-VI). The polarization resistance of coating of test I and test II, after 480 h in SS and 240 h in QCT as per ISO 12944, remain in the range of 10^9-10^{10} ohms.cm^2. This supports the visual observation shown in Table 3. After increasing the exposure period to 1000 h, the impedance decreased considerably with time, however the selected coating system is still in protective range even after extensive exposure which is confirmed by the Nyquist plot (Figure 2a and 2b).
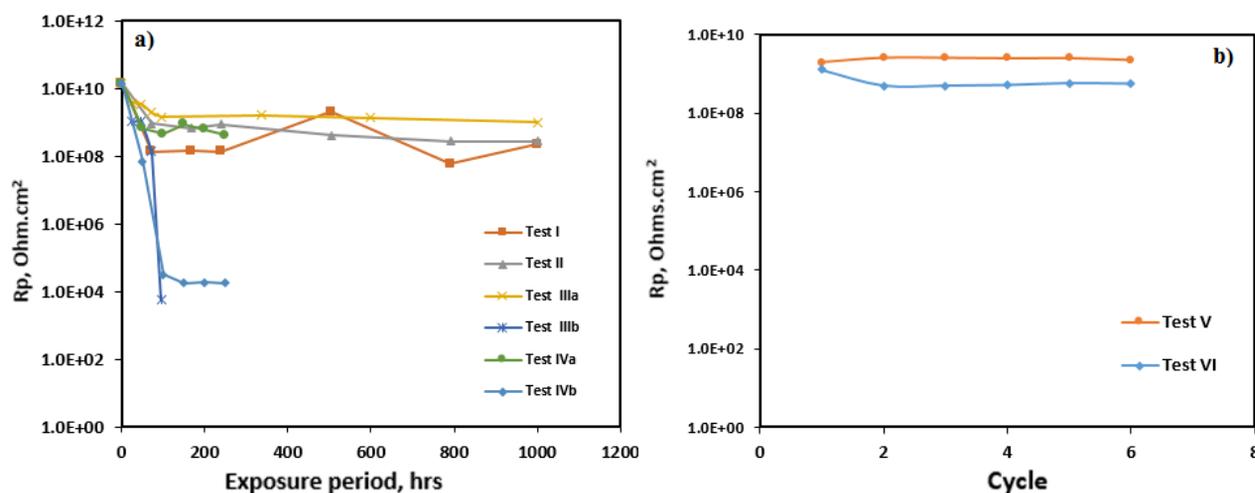
Figure 5: Polarization resistance Vs Exposure time plot (a) test I to IV (b) test V & VI.

Table 3: Corrosion resistance properties of selected WB coating system.

Observation	Performance (ISO 4628)			
	0 = No Corrosion, 5 = Severe Corrosion			
	ISO12944-C3-high		_	
	SS, (480 h)	QCT, (240 h)	SS, (1000 h)	QCT, (500 h)
Blistering	0	0	2	2
Rusting	0	0	2	1
Cracking	0	0	0	0
Flaking	0	0	0	0
Corrosion from scribe	≤1 mm	-	<2mm	-

Bierwagan et al. reported that water penetration causes plasticization of coating, thereby shifts the glass transition temperature [16]. For test condition III (a), coatings remain their protective nature when exposed to distilled water at 50°C for 1000 h. It means there is no severe damage when coating is heated near to glass transition temperature. However, after immersion at 80°C in open environment (test III-b), the coating lost its efficiency to protect the metal in barely 96 h. Organic coating always consists of micro pores. The degree of these micro pores present in the coating varies from polymer to polymer used for coating formulation. It is well known fact that increasing the temperature causes expansion of these micro pores which opens the path for electrolyte penetration. In the case of immersion at 80°C, which is above the Tg, the impedance decreases due to more water uptake and high permeability. This may be the optimum temperature where oxygen has sufficient concentration and temperature is sufficiently high. The combined action of water and high temperature can thus accelerate the coating degradation. It seems that selected WB coating system is more sensitive to the higher temperature than glass transition temperature and this may be the sole reason for failure of the coating.

Under the test condition IV (a) and (b), the coating was immersed in acid and alkali solution for 250 h. As per laboratory practice, any coating which can pass 7 days (168 h) under alkali and acid immersion is considered to be a good protective coating. The coating exposed to acidic solution remains in protective range after 250 h whereas it failed within 150 h in alkali immersion. It shows that coating is not able to retain its protective behavior in alkali environment. This could be attributed to hydrolysis of binder in alkaline condition. In fact, no visual failure (neither blister nor other failure mechanisms) was observed under alkali condition, but EIS showed that the coating lost its protective behavior (Impedance below 10^6 ohms.cm^2) which is also evident from Nyquist plot. This supports the fact that EIS can early predict the failure.

The polarization resistance obtained after completion of 5 cycles in test- V and VI is shown in Figure 5 (b). After completion of 5 cycles, polarization resistance of coating is above 10^6 Ohm.cm^2. It is indicative of superior resistance of coating towards the wet-dry environment thus the coating can tolerate the environment in which sudden changes from wet to dry

condition or vice versa occur. For test condition V, the initial exposure under salt fog at higher temperature (33.3-36.1°C) followed by sudden decrease in temperature (0°C) should give a thermal shock to coating which is followed by humidity at higher temperature (42-48°C) where water vapor penetration can easily deteriorates the coating system. However coating retains its protective behavior after sudden change in environment, suggesting that the coating system is suitable for wet dry environment. In case of test VI, the temperature variation is not able to destroy the protective ability of the coating system. This shows that coating system has the reversible behavior which confirms the good anticorrosion performance.

3.2. Water uptake

The capacitance of coating is a measure of water permeation into the coating. Water has higher dielectric constant than that of polymer, so the capacitance of the coating with absorbed water is higher than that of dry coating. The coating capacitance (Cc) increases at the initial stage of the exposure to an environment. The water uptake of coating in terms of volume fraction of water absorbed (w) was calculated from coating capacitance (Cc) by using Brasher-Kingsbury equation (1) [24].

$$w = \frac{\log\left(\frac{C_t}{C_0}\right)}{\log 80} \tag{1}$$

where, C_t is the coating capacitance after time t and C_o is the initial coating capacitance. The dielectric constant of water is 80. Here, C_0 is the capacitance due to water uptake by the coating before exposure which has been used as standard for comparisons with capacitance obtained at different exposure periods.

The variations in coating capacitance and water uptake for test condition I to VI are shown in Figure 6 (a) and (b). The water uptake under standard test I and II is quite low and steady which is showing that the amount of absorbed electrolyte is not able to accelerate the corrosion phenomenon. This observation supports the higher polarization value for coating system under these test conditions. Among all accelerated exposures, the lab designed test III (b) shows highest water uptake. It appears that at high temperature, film has low resistance and fine pathways for easy ion transport which enhances the penetration of the water in to the interior film.
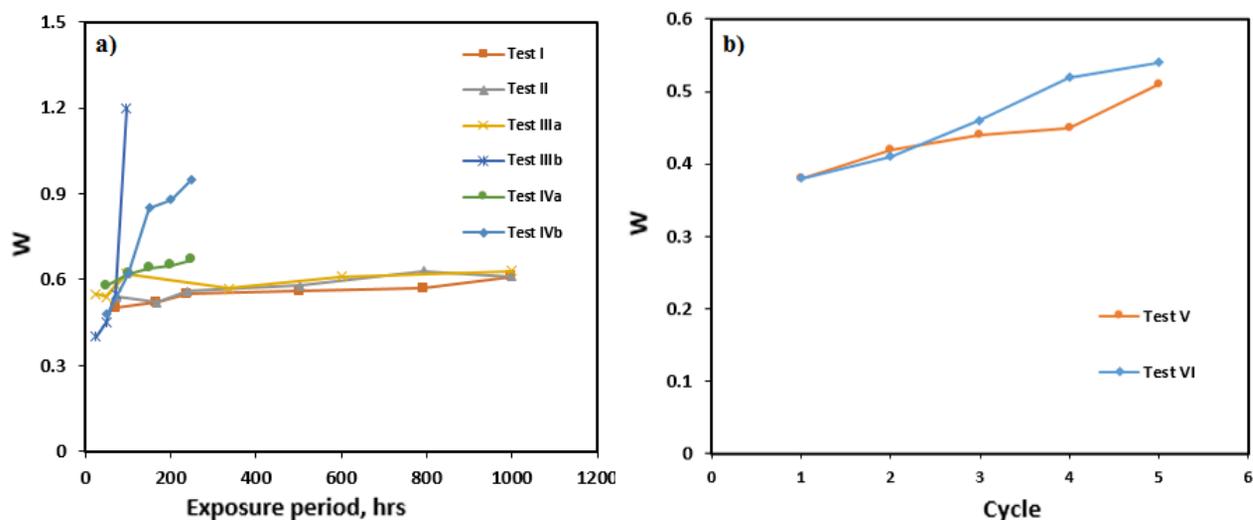
Figure 6: Water uptake of coatings with exposure time (a) For test I to IV and (b) test V and VI.

The penetration of water causes swelling of the polymer particles close to the interface. This leads to the higher water uptake values. Whereas, in case of test V and VI, there are limited pathways for water penetration and absorption which result in lower water uptake. It is evident from the results that heating the coating system above glass transition temperature leads to high deterioration of the coating.

3.3. Breakpoint frequency (f_{45})

The breakpoint frequency method presented by Haruyama et al. [25] permits measuring the delamination area of polymer coatings on steel in corrosive environment. The breakpoint frequency is defined as the frequency at which the phase angle reaches 45^0. The breakpoint frequency was determined from phase angle Vs $\log f$ plot at $45°$. The mathematical expression for breakpoint frequency is

$$f_{45}=K\frac{A_d}{A} \tag{2}$$

In which, the ratio of delaminated area or reactive area (A_d) and the total area of the sample (A) are proportional to the breakpoint frequency (f_{45}). K is considered to be a material constant for the coating dependent on the system under study. Since the tested area of all the samples is constant, breakpoint frequency will be proportional to reactive area or delaminated area.

$$f_{45} \; \alpha \; A_d \tag{3}$$

It can be seen from the Figures 7 (a) and (b) that the breakpoint frequency of the coatings increases with exposure period for all test conditions. The breakpoint frequency under standard test conditions (test I and II) is lower than that under lab designed test conditions. The higher breakpoint frequency value for test III (b) and IV (b) indicates faster deterioration of the coating. The highest f_{45} value for test III (b) shows that maximum reactive area among all other test conditions is exposed. The reactive area is the area where electrolyte can penetrate and accelerate the corrosion. This directly indicates that the coating system is losing its ability to protect the metal which is the reason that more electrolytes are penetrating. The breakpoint frequency value for both test conditions are in good agreement with their lower polarization resistance.

Breakpoint frequency for coating system under test-V after 5 cycles did not increase significantly (Figure 7b). It indicates that little extent of area is exposed for corrosion, and thus higher polarization resistance value was recorded. The similar kind of observation was found for test-VI. It implies that the exposure area for corrosion is less in this case, which shows that coating has the power to regain its protection ability even after thermal shock. This also support to higher polarization resistance for the selected coating system under this test condition.

3.4. Adhesion strength

Adhesion strength of anticorrosive coating before and after exposure to different accelerated test conditions (I to VI) is shown in Figure 8. It can be seen from the results that adhesion strength of the anticorrosive coating decreases after exposure to most of the tests. This is attributed to the penetration of water through coating during exposure leading to weakening of hydrogen bonding between metal oxide and the coating interface. Test III (b) and IV (b) showed the loss of adhesion and that the under-film corrosion has occurred at higher rate. The results obtained for adhesion strength are further strengthening our earlier finding obtained by EIS study that reduced polarization resistance of test III (b) and IV (b) compared to others. The results showed that the coating exposed to hot water at 80°C showed faster rate of degradation compared to other conditions.
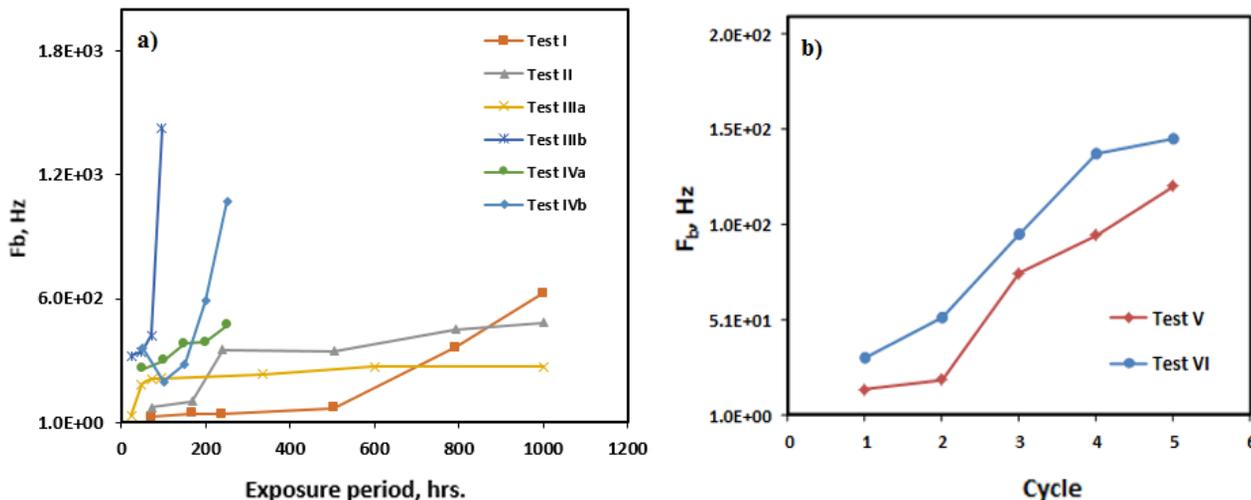
Figure 7: Breakpoint frequencies of coatings under various conditions (a) test I-IV and (b) test- V and VI.
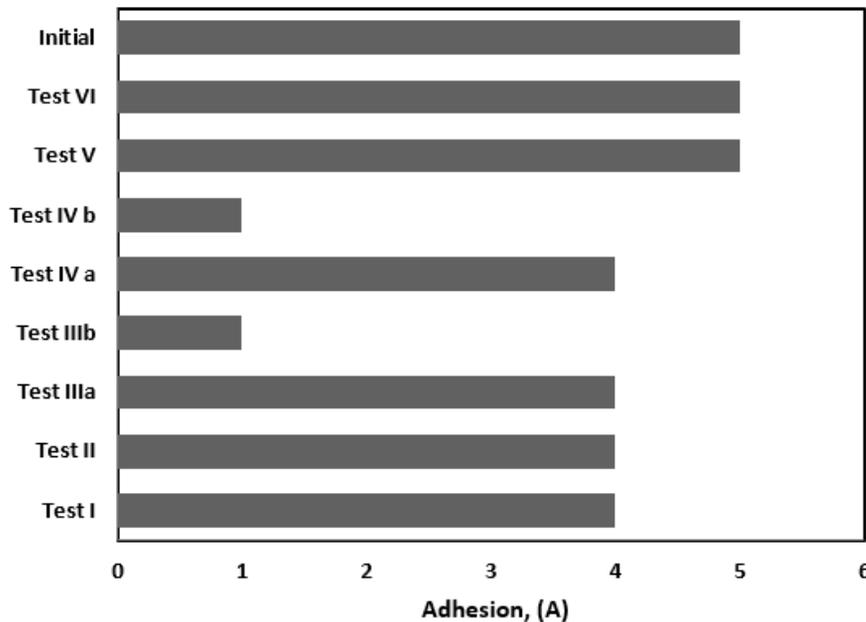
Figure 8: Adhesion strength of exposed coatings to different test conditions.

4. Conclusion

Waterborne Epoxy-PU coating system, according to ISO 12944 C3 High, exposed to neutral salt spray and QCT shows polarization resistance well above the 10^6 ohm.cm^2, even after 1000 h. Early failure of coating in the case of chemical immersion test indicated poor resistance property of coating against chemicals. The results of polarization resistance (Rp), water uptake (w) and break point frequency (f_{45}) obtained under all accelerated exposure conditions were in agreement with each other. Among all accelerated exposures, deterioration of the coating was faster when the coating was exposed to elevated temperature (80°C) in distilled water. Residual adhesion strength after completion of each test also confirms these observations.

The results showed the response of selected coating system under different exposure conditions and gave the insight about the behavior of the coating under different failure modes. It suggests that coating which passes ISO 12944 C3 (high) performance requirement is not sustaining in distilled water immersion at 80°C for barely 96 h. In other words, we can assume that the coating which can pass even 96 h in distilled water immersion (80°C) can pass the required performance for ISO 12944 C3 (high) environment. However, more experimentation with different coating systems will be needed for stabilizing any significant correlation.

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