The Study of Corrosion Behaviour of Intumescent Fire Retardant Coating with Structural Steel Substrate

W. C. Puspitasari[1], Faiz Ahmad[1,*], Sami Ullah[2], M. Rafi Raza[3], P. Hussain[1], P.S.M. Yusoff[1] and Yasmin, Azmi[1]

[1] Mechanical Engineering Department, Universiti Teknologi PETRONAS Bandar Seri Iskandar, 32610, Perak, Malaysia.
[2] Department of Mechanical Engineering, University of Gujrat, 50700, Pakistan.
[3] Department of Mechanical Engineering, COMSATS Institute of Information Technology, Sahiwal 57000, Pakistan
*E-mail: faizahmadster@gmail.com

The aim of this work is to study corrosion behaviour of intumescent coating and the primer on steel substrate before and after a fire test. The coated steel substrates were subjected to fire in a furnace at 950°C for a variable duration ranging from 30, 45, 90 and 120 min. All coated steel substrates were also subjected to corrosion test by immersing them in 5% NaCl solution, for 15 min and three months duration using Electrochemical Impedance Spectroscopy (EIS). The specimen's impedance before fire test and before immersion was $4.37 \times 10^8 \Omega.cm^2$. After 15 min exposure to NaCl, this value was reduced by 26, 53, 60 and 65% for specimens fired for 30, 45, 90 and 120 min, respectively. Further reduction in the impedance by 97.6, 97.8, 97.9 and 98.9% for samples fired for 30, 45, 90 and 120 min, respectively, were measured after three months in 5% NaCl immersion.

1. INTRODUCTION

Due to the technological growth of the large-scale industry, the environment with acidic atmospheric has become the major issue. The precipitations of the acidic environment are formed when particular contaminants such as sulfur dioxide (SO_2) and nitrogen oxides (NO_X) are released into the environment and further carried by wind and air currents into the atmosphere. In the atmosphere, these contaminants combine with water, oxygen and dust particles to form sulphuric and nitric acids. In addition of these two main acidic contaminants, the burning of the coal HCl is formed as a by-product and is also found in the environment [1].

Steel has an important role in the construction of offshore platforms, marine, and furniture industry. Fire protective materials are used to reduce the overwhelming cost of fire in terms of property damage and loss of life while applied in building structures [2,3]. Steel has recrystallization temperature 500˚C and above, at higher temperature the rapid recrystallization occurs which destroy the ferrites structure. When a building catches fire and the temperature reaches 500˚C and above and it leads to the structural collapse [4]. According to the building regulations (HMSO, 1992) implemented in the United Kingdom, Europe, and the USA, in case of fire emergency, ensure the safe evacuation of people from the building [5-7].

Under severe fire conditions, intumescent coatings have the ability to retain the steel integrity between 1 to 3 hours even the surrounding temperature reaches 1100 °C [8-10]. Intumescent fire retardant coatings are widely used to reduce the flammability and to maintain the integrity of the steel structures. Intumescent coatings have three main advantages: prevent the penetration of heat, spreading of flame and does not modify the intrinsic properties such as mechanical steel coated with the intumescent coating [11]. The corrosion behaviour of intumescent coatings with steel substrate is not fully discussed in details in the literature [12-16]. The reactions of the intumescent coating occur during combustion at high temperatures. The reactions can occur in vapor, the condensed phase or both. The combustion of the intumescent coating is a complex process and there may be different mechanisms occurring as the combustion of ingredients increases.

Several researchers investigated the durability of the coatings [17-19]. However, the research on the longstanding fire resistant to fire retardant coatings is comparatively limited. Jimenez et al. studied the degradation of an intumescent coating which comprises melamine, epoxy resin, titanium dioxide and ammonium polyphosphate in accelerated aging conditions such as 80% moisture atmosphere at 70 °C for 2 months. Furthermore, they used a static immersion bath with and without NaCl (5 g/L) at 20 °C for 1 month) [20]. Ullah et al. studied the effect of kaolin clay in the intumescent coating in an accelerated weathering chamber according to ASTM D 6695-03. They reported that the physical appearance of the coatings were changed from light black colour to light yellow and the char expansion after fire test was also reduced compared to non-weathering test [2].

The objectives of the current study are to investigate the effect of salt spray and corrosion of the durability of the intumescent coating to the steel substrates.

2. MATERIALS AND METHOD

2.1. Materials

The commercial intumescent coating and the primer coating were supplied by International Paint Sdn Bhd Pasir Gudang, Johor Akzo Nobel Malaysia. The commercial coating is reinforced epoxy intumescent fireproofing, suitable for the protection of steel, aluminum, and others from cellulosic and hydrocarbon (pool and jet) fires. A good primer coating is the one which satisfies its qualification procedure from International Coatings. Intergard 251 is zinc phosphate based primer coating used in this study. A two-component epoxy, anti-corrosive primer pigmented with zinc

phosphate was used on the steel substrate. The detailed chemical composition of the coating and primer is given in Table 1 and 2, respectively.

Table 1. Intumescent coating resin and hardener composition

Ingredients	Conc. range (wt.%)
PART A	
Epoxy resin (av.mol.wt.<700)	25 - < 50
Triphenyl phosphate (TMP)	2.5 - < 10
PART B	
Tetraethylenepentamine	1 - < 2.5

Table 2. Primer coating composition

Ingredients	Conc. range (wt.%)
PART A	
2-Methoxy-1-propanol	<1
Bis-amine wax	<1
Epoxy Resin liquid	10-25
Ethyl Benzene	2.5-10
Iron oxide	2.5-10
Propylene glycol monomethyl ether	2.5-10
Talc	10-25
U.F. Resin	<1
Xylene	10-25
Zinc phosphate	2.5-10
PART B	
2,4,6-Tris(dimethylaminomethyl)phenol	2.5-10
Xylene	>50

2.2. Steel Substrate Preparation

For the preparation of the steel substrate, the surface of the steel samples was cleaned to remove grease, dirt, and oil using lacquer thinner and its chemical compositions consist on toluene (toluol), xylene (xylol), methyl ethyl ketone. After cleaning the surface of the steel samples, sandblasting was performed using Air Compressor Airman 370 CFM. Garnet sand with particle size 30-60 mesh and 20-40 mesh were shot from nozzle size 1.8 mm for about 10 minutes to each steel surface. The steel substrate surface roughness was achieved 50-55 um, according to surface preparation standard NACE International SSPC-SP10 [21]. MOHR Profilometer was used to measure the surface roughness with testing distance 12.5mm and an average of three measurements is reported in this study. This equipment measured an average surface roughness by comparing all the peaks and valleys to the mean line and then averaging them all over the entire cut off length. Ra is general surface roughness while Rz is mean roughness depth.

2.3. Intumescent Coating Preparation

In this study, steel samples were coated with primer layer of I mm thickness and a 5×5 cm^2 sheet of carbon fiber is placed on the steel substrate. The primer was allowed to dry for 6-7 hours at room temperature and complete dryness was ensured by touching prior the intumescent coating was applied. The success of primer coating fulfill the jagged interface on steel depends on the surface tension of primer coating and steel substrate. Flaws and dirt would increase the surface tension on steel surface and result in poor adhesion of primer coating. To eliminate the flaws on the jagged steel surface, good wetting of the surface is important. Intimate contact occurs when the adhesive spontaneously spreads over the surface. Spreading (spontaneous or not) can be examined by contact angle measurement [22, 23].

Mixing of correct proportion of epoxy intumescent coating and hardener was performed as ratio by weight of part A (resin) to part B (hardener) was 2.45: 1 and 4: 1 for intumescent coating and primer coating, respectively. A small amount of thinner as a solvent was added to the mixture to improve the flowability to be applied to the steel substrate. This solvent evaporated and did not affect the coating properties. The coatings were prepared by mixing epoxy and hardener using a mixer at 600 rpm. After the homogenous mixture was achieved, this mixture was applied to the steel substrate surface (5 x 5 cm^2). The thickness of 10 mm of intumescent coatings on the steel substrate was studied in this work. The thickness of the coating was measured by digital vernier calliper before and after dry.

3. CHARACTERIZATION

3.1. Heat Insulation Test

The fire test was carried out for 30, 45, 90 and 120 minutes. The heat of the fire was set up according to hydrocarbon fire temperature. The fire was shot from Liquefied petroleum gas (LPG) Bunsen burner with a temperature of 950°C at 7cm from the intumescent coating surface. The steel substrate surface was monitored every second for 300 seconds and the temperature was measured using Hanna Thermocouple. The thermocouple was attached to the backside of steel substrate to measure the temperature on the sample surface.

3.2. Salt Spray Test on the Coated Substrate

The chemical resistant of the coating was measured using Salt Spray test chamber according to the required standard ASTM B117 [24]. Two types of samples, the bare steel substrate, steel substrate coated with primer and intumescent coating were sprayed with 5% NaCl solution for 2 weeks at 35°C.

3.3. Electrochemical Impedance Spectroscopy (EIS) Test

The corrosion resistance at the interface of coating with steel substrate is important to be investigated. Electrochemical impedance spectroscopy (EIS) is usually used to investigate the corrosion resistance of coated metal in various corrosive environments. The EIS measurements were performed at the open circuit potential (OCP) in 5% NaCl solution, repeatedly for different exposure periods, using a frequency response analyser. The EIS was measured in the frequency range $0.5\text{-}10^5$ Hz, using perturbation amplitude of 20 mV. The EIS data were analysed by using the Z-View software.

An electrochemical cell is a device assisting the chemical reactions to generate electrical energy. During the chemical reactions, the electrons discharge from one substance to another, due to redox reaction. The substance which oxidized and lose electrons is called reductant; on the other side, the oxidant is the species which is reduced in the process by gaining electrons. The related potential energy can be resolved by the potential difference between the valence electrons in atoms of different substances.

The electrochemical tests were implemented out in a three-electrode (each with surface area 1 cm^2) corrosion cell for the quantitative examination of coating corrosion. The working electrode is the coated steel sample which is intended to be observed and to measure the corrosion rate. The counter/auxiliary electrode is the name specified for the second electrode which precisely transmits the current in the circuit formed by the observations. The third electrode was an Ag|AgCl, 3.5M KCl electrode as a reference.

The corrosion resistance measurement was performed by measuring the impedance of the coated sample with graphite as an auxiliary electrode and Ag/AgCl as the reference electrode. The set up was made of a Gill 12 Weld tester ACM machine with an ACM Instrument analyser. The electrolyte solution was 5% NaCl, made from 5g of 99.99 wt. % purity NaCl in 100ml of distilled water. The pH of the solution was 6 and the temperature was 25°C. Every sample tested was connected with copper wire and mounted via cold mounting. The surface of the sample was flat and clean from dirt, oil or dust before the test. The surface of the steel substrate samples (before and after fire test of the coating at a different time) was ground using SiC paper with grid 120-800, and then it was cleaned with water and ethanol to remove all the dirt. The coating quality of the sample before fire test, after fire test and after three months' immersion was tested using EIS test. The adhesion bonding condition also can be measured by the test. Three months' immersion was chosen as an average time allowance for repair the structure.

3.4. Microstructure Examination

The morphology of the interface layer of the steel substrate and the interface layer between the primer coating and the intumescent coating was observed using Scanning Electron Microscope (SEM) Oxford Leo 1430.

4. RESULTS AND DISCUSSION

4.1. Heat Insulation Test

Figure 1 shows coated sample before and after fire test. Under the fire test, when the coating attains its critical temperature, it begins to melt and converted into a liquid. Simultaneously, initiated reactions release inert gases which trap within the char resulting causing the reduction in its thermal conductivity [25]. These gases were trapped inside the viscous fluid (formation of bubbles).
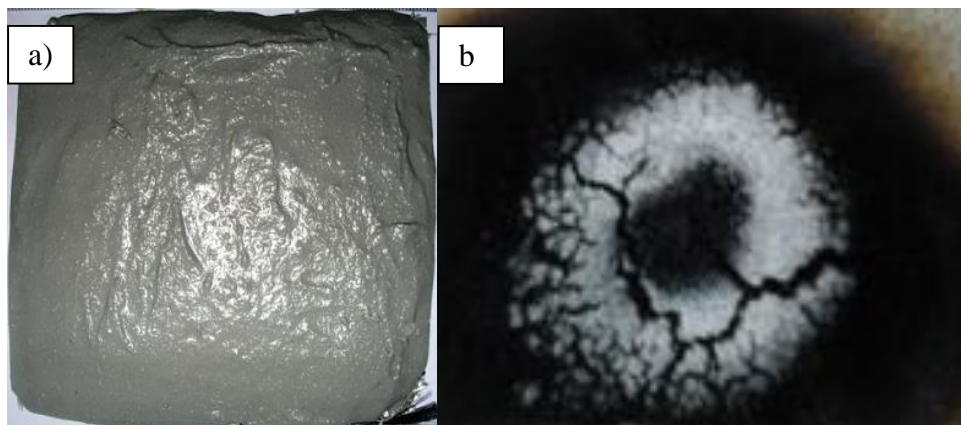

Figure 1. Intumescent coated steel test sample, a) before fire test and b) after fire test.

During the fire test, the coating expands several times of its original thickness (2-10mm) and resulted char behave as an insulative barrier between fire and substrate (Figure 1b) [26]. The thickness of the formed char is 4 times higher than the original thickness of the intumescent coating. The thick char has a much lower thermal conductivity than in the intumescent coating before its burning. The constant substrate temperature showed that the coating provided an excellent insulation with the formation of the multi carbonaceous char layer during the two-hour fire test and the low hardness of intumescent coating can improve the presence of various additives in the material.

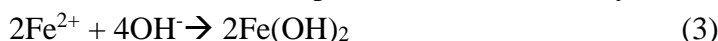### 4.2. Salt Spray on Steel Substrate

Figure 2(a) shows the steel substrate before salt spray. Figure 2(b) shows that oxide fully covered the surface of steel substrate after salt spray test with 5% NaCl concentration exposed for two weeks. A simplified chemistry of a corroded iron piece is summarized in Eq. 1 to 4 [27]:
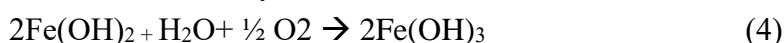
$Fe \rightarrow Fe^{2+} + 2e^-$ (anodic reaction) $\qquad$ (1)

Water and oxygen area basic requirements to initiate corrosion

$2H_2O + O_2 + 4e^- \rightarrow 4OH^-$ $\qquad$ (2)

Formation of corrosion product is the iron(II) hydroxide

$2Fe^{2+} + 4OH^- \rightarrow 2Fe(OH)_2$ $\qquad$ (3)

Later on, iron (II) hydroxide was oxidized and formed iron(III) hydroxide, or ferric salt.

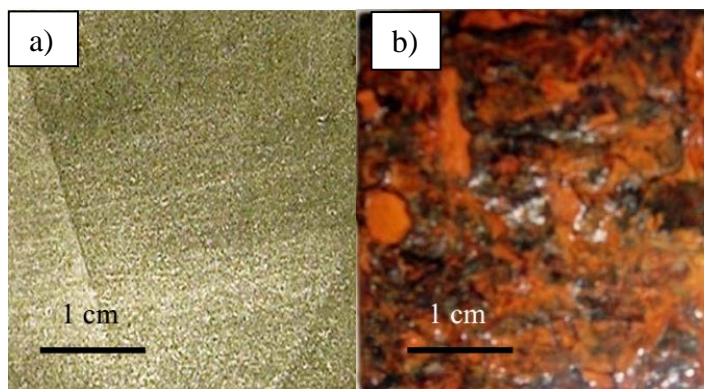$2Fe(OH)_2 + H_2O + ½ O2 \rightarrow 2Fe(OH)_3$ $\qquad$ (4)

Figure 2. Steel substrate was sprayed with 5% NaCl, a) before salt spray b) after salt spray

The copious corrosion product on steel substrate shows that low carbon steel has poor corrosion resistance. The real chemistry of the corroded iron and steel is complicated and somehow embraces the field of chemistry and electrochemistry. Iron corrodes only in the presence of both water and oxygen.

4.3. Effect of Salt Spray on Steel Substrate Coated with Primer Coating

Figure 3(a, b) shows the micrographs of steel substrate coated with zinc phosphate as a primer before and after salt spray test, respectively. Figure 3(a) shows the zinc phosphate primer coating that was applied to the steel substrate surface before salt spray test. According to Figure 3(b), the corrosion product was formed starting from the uncoated side it might be due to the back side of the plate was not fully covered with primer during the salt spray. It indicates that the steel still can be corroded unless the primer coating uniformly covered the surface and no defect in the coating.
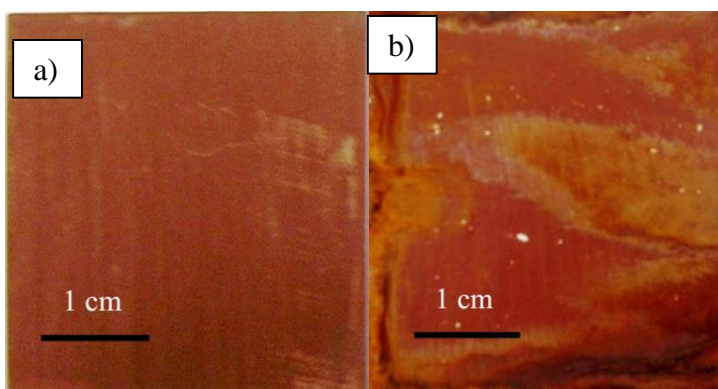

Figure 3. Steel substrate coated with zinc phosphate primer coating, a) before salt spray test, b) after salt spray

4.4. Effect of Salt Spray on Steel Substrate Coated primer and Intumescent Coating

Figure 4(a, b) shows the samples of intumescent coated steel before and after salt spray. The intumescent coating was applied to the surface of primer coating and steel substrate. The epoxy-based intumescent coating is known to have excellent corrosion properties since epoxy has hard and durable structure. Figure 4(a) shows the surface of the intumescent coating was uniform and no defect on the surface. The 5% NaCl was sprayed for two weeks to the coated sample and the result is shown in Figure 4(b). The figure shows that there is no corrosion product was produced on the surface after the exposure to salt spray. This is because the electron cannot easily transfer to the coating structure due to the crosslinked structure of epoxy.
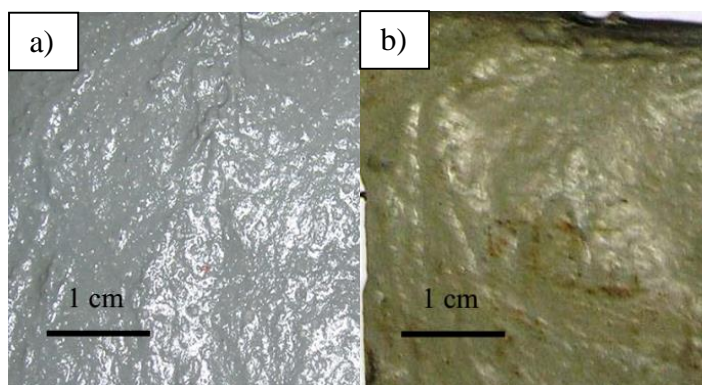

Figure 4. Intumescent coated Steel substrate, a) before salt spray b) after salt spray

4.5. Electrochemical Impedance Spectroscopy (EIS)

The effects of fire test duration on adhesion bonding between steel substrate and the intumescent coatings were revealed by EIS test. Measurements were performed in the frequency range of 0.5 Hz to 10^5 Hz. The Nyquist graphs of NaCl immersion for 15mins and 3months flowed by the different fire exposure time are shown Figures 6 and 9 respectively. The respective bode plot and phase angle plot are presented in Figures 7, 8, 10 and 11, respectively. The summary of polarization resistance and rest potential are given in Table 3 and 4 before and after immersion with fire test durations, reactively.

4.5. EIS after fire test

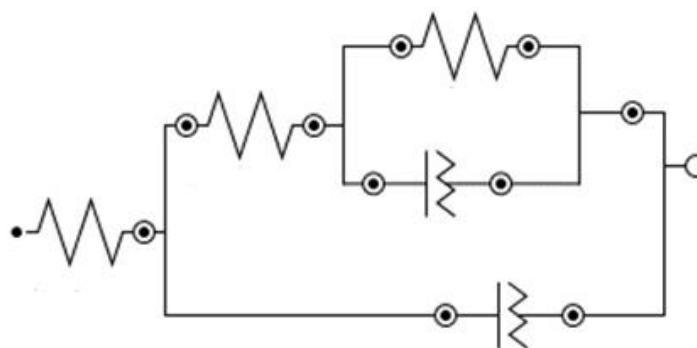Figure 5 represents an equivalent circuit model of fitting EIS data. Figure 6 shows a Nyquist graph of intumescent coated steel before and after fire test. According to theory, the coating impedance values higher than $10^7 \Omega.cm^2$ indicate that the coating has excellent corrosion resistance while coating impedance between 5×10^5 to $10^6 \Omega.cm^2$ indicates the area of steel to be corroded is increased from 0.3% to 1 % [28-30]. The coating impedance before fire test (0 min) is $4.37 \times 10^8 \Omega.cm^2$. The results showed that the impedance valued decreases to $1.52 \times 10^8 \Omega.cm^2$ with fire exposure time 120mins.

However, the achieved value impedance of coating is in the range where it can withstand. The degradation most probably happens because a thicker char was formed by increasing the time, hence the cross-linked structure of epoxy was broken, thus water easily penetrated to the steel substrate [31]. The summary of polarization resistance and rest potential are given in Table 3. Bode plot and phase angle plot of EIS spectra systems of the coating resistant after fire test is illustrated in Figure 7 and 8, respectively.

Figure 5. Representing an equivalent circuit model of fitting EIS data
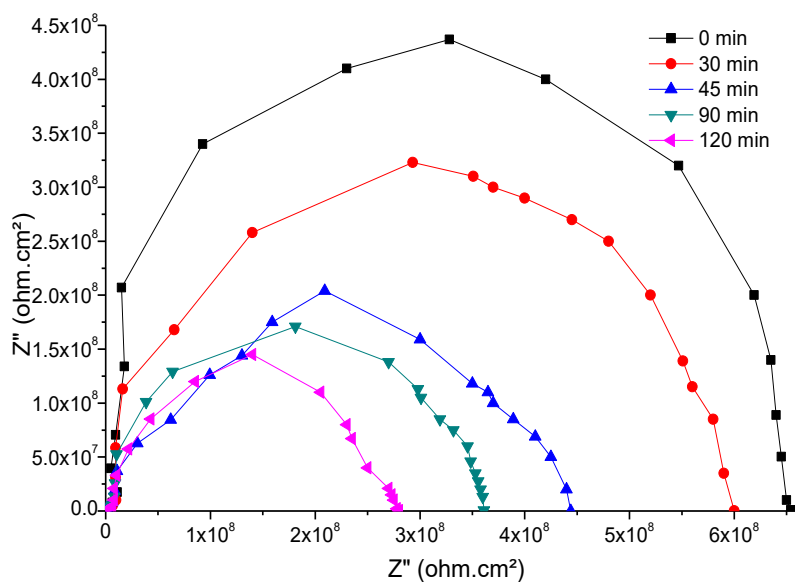

Figure 6. Nyquist plot of intumescent coated steel sample before and after 15 min exposure to NaCl exposure for 30 to 120 minutes of fire test.
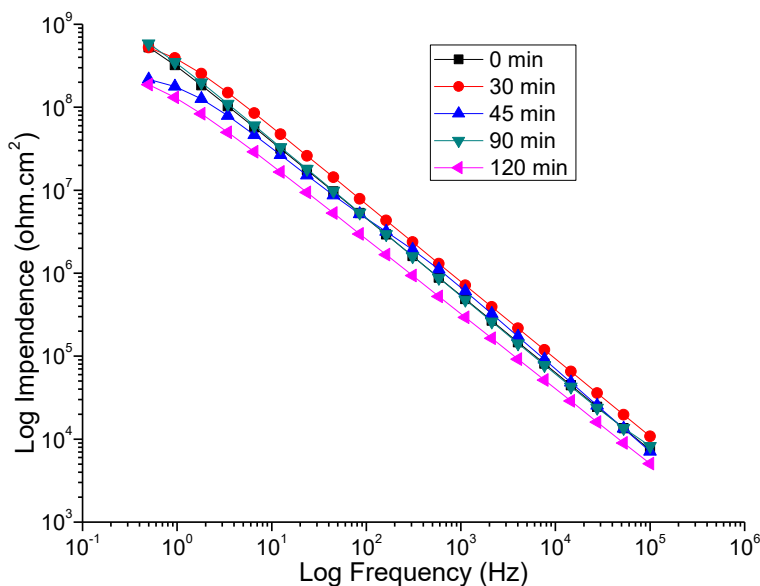
Figure 7. Bode plot of EIS spectra systems of the coating resistant after fire test exposed to 5% NaCl solution 15 min; solid lines are the fitting curves with equivalent circuits.
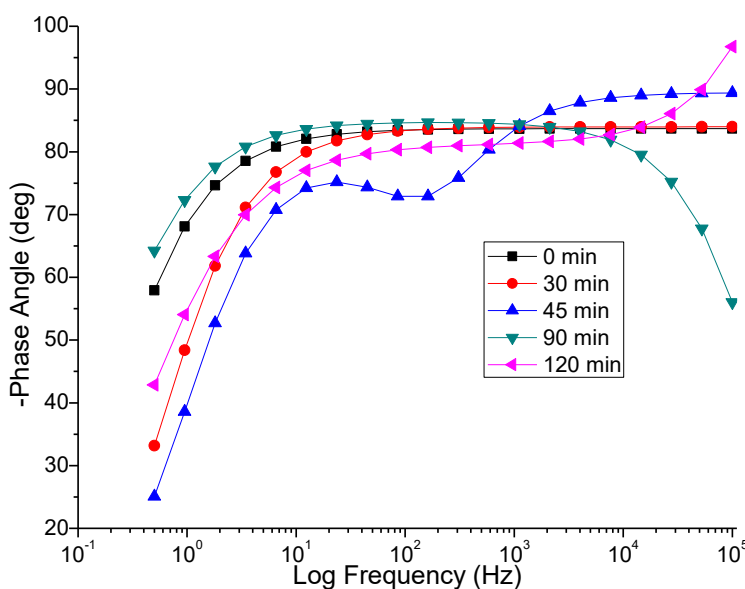

Figure 8. Phase angle plot of EIS spectra systems of the coating resistant after fire test exposed to 5% NaCl solution 15 min; solid lines are the fitting curves with equivalent circuits.

Table 3. EIS observation of coating before immersion with fire test duration

Immersion time(min)	Before Immersion						
	Rs	Rc	Q1	N	Rpo	Q2	N
0	0		5.46E-10	0.92996	4.00E+08	2.11E-09	0.29267
		1.02E+09					
30	0	5.00E+06	3.60E-10	0.93307	6.67E+08	4.55E-13	0.6731
45	0	6.10E+06	2.44E-10	0.9939	2.51E+08	4.80E-10	0.86023
90	4000	1.54E+09	4.97E-10	0.94275	1.10E+12	1.79E-09	0.8
120	-1366.9	-1.10E+12	1.15E-09	0.90273	1.10E+12	1.15E-15	0.61745

The results showed that before fire test at 0.5Hz the coating impedance was 4.46×10^8 $\Omega.cm^2$ and this value was reduced to 5164.2 $\Omega.cm^2$ at 10^5 Hz. A similar pattern was observed after the fire test. However, by comparing the impedance value with the coated steel, small decrease in impedance was observed by increasing the fire exposure time. The intensity of peak reduced with exposure time indicates that the less capacitive response; indeed, as the duration of the fire time increases.

4.6. EIS after three months' immersion in NaCl

The Nyquist plot in Figure 9 shows that the coating impedance value is dramatically decreased to 1.43×10^7 $\Omega.cm^2$, 1.03×10^7 $\Omega.cm^2$, 9.55×10^6 $\Omega.cm^2$, 8.94×10^6 $\Omega.cm^2$, $4.52 \times 10^6 \Omega.cm^2$ after three months immersion in NaCl solution for sample before fire (0 min) and after fire test for 30, 45, 90 and 120 minutes. This may be due to the absorption of NaCl solution by the coating resulted in the degradation of chemical properties of the intumescent coating, primer coating, and steel substrate gradually degraded and lost the corrosion resistance after applying the heat to the coating surface [32]. The EIS results in Figure 9 reveal that the higher quantity of water uptake in the intumescent coating and primer coating reduced the impedance of the coating and resulted reduce the quality of the coating. The significant reduction of impedance after immersion is believed that the corrosion occurred on the steel during immersion [33]. Bode plot and phase angle plot of EIS spectra systems of the coating resistant after fire test is illustrated in Figure 10 and 11, respectively.
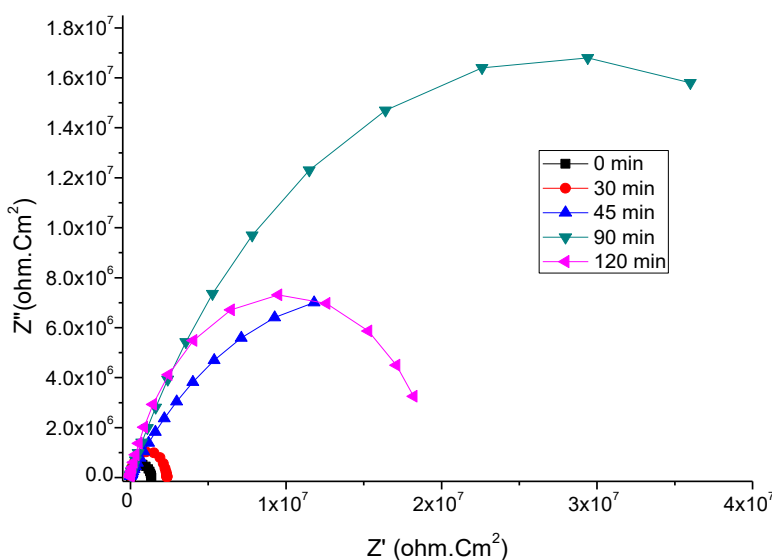

Figure 9. Nyquist plot of intumescent coating resistance of before and after fired samples for three months' immersion in 5% NaCl.

Table 4 shows the polarization resistance and rest potential of the samples. The reduction of polarization resistance and rest potential indicates the resistance of steel substrate to be corroded is

increased. The primer coating was getting weaker to protect steel from corrosion by increasing the fire test duration and also immersion in the electrolyte solution.

Table 4. EIS observation of coating after immersion with fire test duration

Immersion time(min)	After Immersion						
	Rs	Rc	Q1	N	Rpo	Q2	N
0	21480	1.33E+06	2.65E-09	0.85871	0	0	0
30	-16387	1.17E+06	5.46E-10	0.91882	1.19E+06	3.22E-12	0.98465
45	-38951	-1.43E+08	2.45E-08	0.57493	1.73E+08	9.06E-15	0.83742
90	-46435	-1.07E+12	5.21E-09	0.69316	1.07E+12	5.99E-15	0.43274
120	-6070.8	1.63E+07	3.94E-09	0.80643	3.60E+06	1.91E-12	1.1

The decrease in coating resistance also shows that the electrolyte solution was contacted with the steel substrate and corrosion is initiated. The SEM micrograph (Figure 12) on the interface of primer coating and steel shows a narrow gap was created and also the degradation of primer steel occurred at the time.
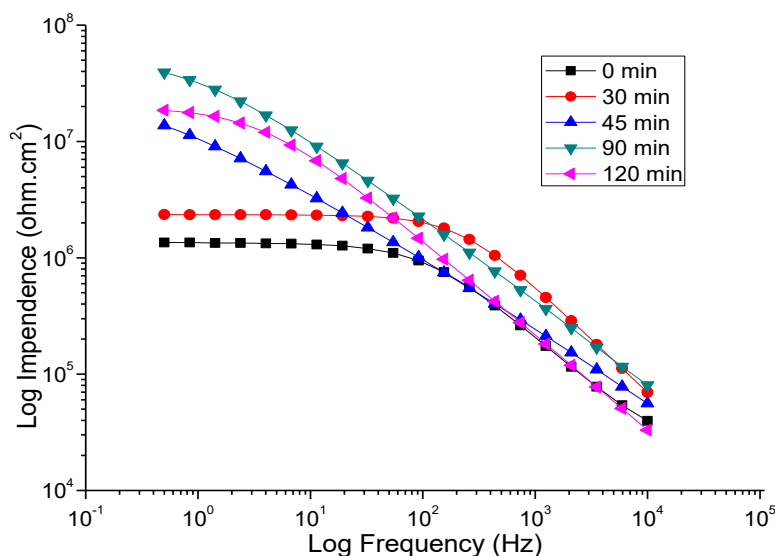

Figure 10. Bode plot of EIS spectra of the coating resistant exposed to 5% NaCl solution 3 months immersion
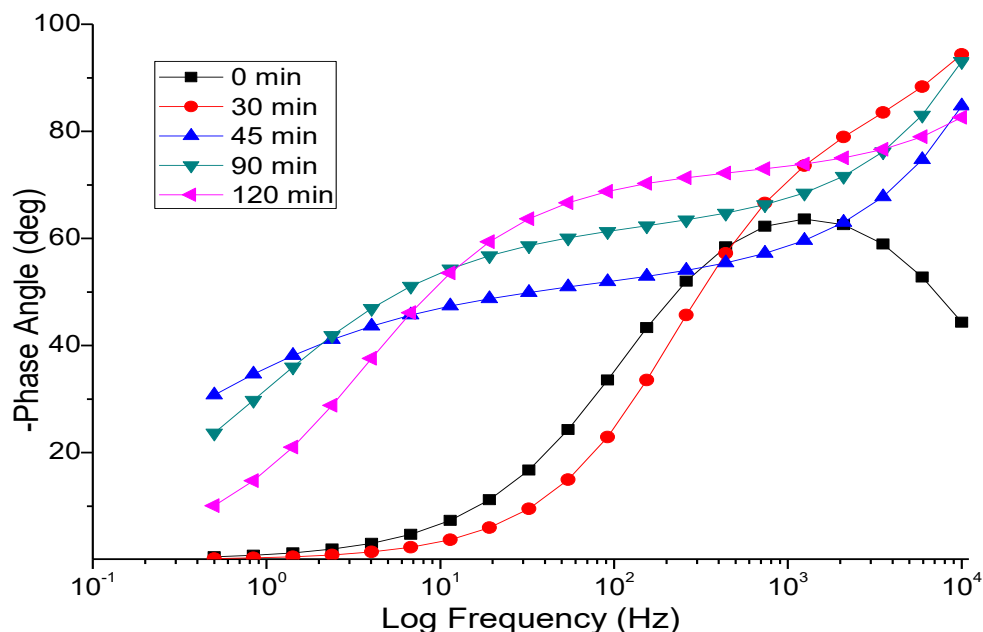
Figure 11. Phase angle of EIS spectra of the coating resistant exposed to 5% NaCl solution 3 months immersion

This gap could be a corrosion site since corrosion only happened due to the presence of oxygen and water. Bode plot and phase plot of EIS spectra systems of the coating after 3 months immersion resistant after fire test is illustrated in Figure 10 and 11, respectively. The results showed that before fire test at 0.5Hz the coating impedance was 2.10×10^6 $\Omega.cm^2$ and at 10^5 Hz it was noted 7.05×10^4 $\Omega.cm^2$. After fire test, the impedance was reduced to 1.05×10^6, 9.66×10^3, 1.0368×10^4, 1.00×10^4 $\Omega.cm^2$ at 0.5 Hz for 30, 45, 90 and 120 min fire test duration, respectively. At the frequency 10^5 Hz the impedance for the steel samples was drastically decreased to 4.02×10^4, 6.06×10^3, 4.41×10^3 5.24×10^3 $\Omega.cm^2$ of 30, 45, 90 and 120 min fire test duration, respectively. Figure 12 (a, b) shows the electron micrographs of two hours exposure with fire test before and after three months immersion sample in 5% NaCl solution.

Figure 12(a) shows the surface degradation of intumescent coating and primer coating after fire test might be the reason for the decreasing of the coating performance. The arrows indicate the coating structure has failed even before immersion in the electrolyte solution. The evidence of crack of the coating and the corrosion occurred after three months' immersion in NaCl is shown Figure 12(b). The dramatic decrease in impedance is due to the primer coating failed to protect the steel by letting the steel surface to be corroded. This Figure also shows that the jagged surface could be a high-risk location for pitting corrosion. The three months' immersion in NaCl proves that water uptake could break down the adhesion bonding between intumescent coating, primer coating, and steel substrate. The resistance of the coating changes during exposure due to the penetration of electrolyte into the microspores of the coating [34].
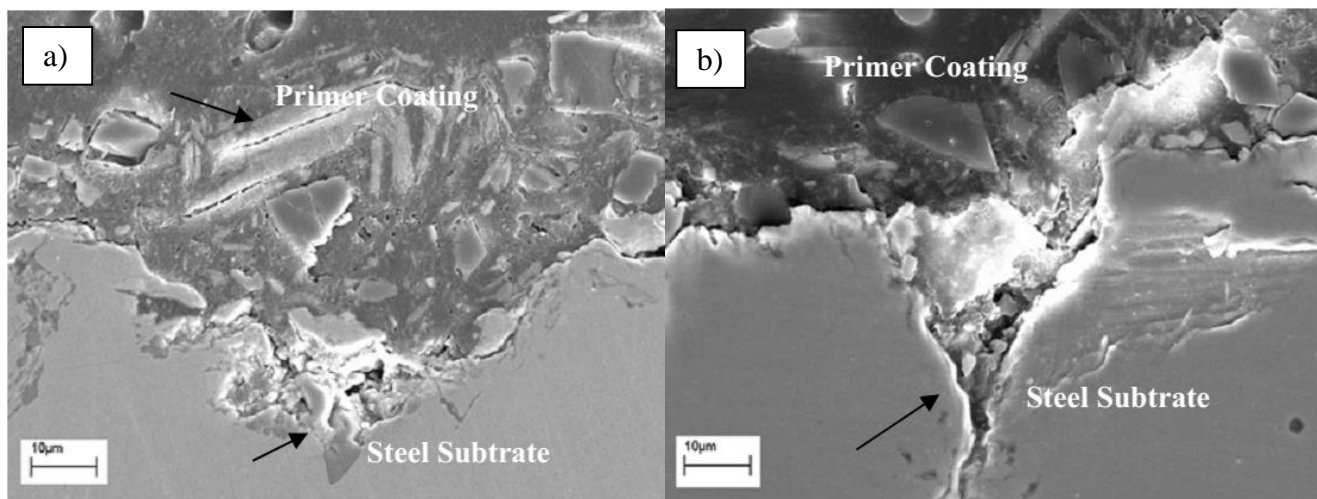
Figure 12. SEM micrographs of the intumescent coated sample, a) before immersion and b) after 3 months immersion in 5% NaCl.

5. CONCLUSION

The developed coating was successfully tested for fire test and corrosion resistance for various duration ranging from 30, 45, 90 and 120 min. The coating impedance before fire test was 43.7 x $10^7 \Omega.cm^2$. After 15 min exposure to NaCl, this value was reduced by 26, 53, 60 and 65% for specimens fired for 30, 45, 90 and 120 min, respectively. Further reduction in the impedance by 97.6, 97.8, 97.9 and 98.9% for samples fired for 30, 45, 90 and 120 min, respectively, were measured after three months in 5% NaCl immersion.

References

1. W. Rodgers, D. Garner, G. Cheever, *J. Coat. Technol.*, 70 (1998) 83.
2. S. Ullah, F. Ahmad, A.M. Shariff, M.A. Bustam, *Polym. Degrad. Stab.*, 110 (2014) 91.
3. M. Zia-ul-Mustafa, F. Ahmad, S. Ullah, N. Amir, Q.F. Gillani, *Prog. Org. Coat.,* 102, Part B (2017) 201.
4. A.V. Sverdlin, A.R. Ness, Steel Heat Treatment Handbook, Marcel Dekker, Inc (1997) New York, United States.
5. F. Wald, L.S. da Silva, D. Moore, T. Lennon, M. Chladna, A. Santiago, M. Beneš, L. Borges, *Fire Saf. J.*, 41 (2006) 509.
6. P. Hughes, E. Ferrett, Introduction to Health and Safety at Work: For the NEBOSH National

General Certificate in Occupational Health and Safety, Routledge, (2015) New Yourk, United States.

7. P. Hughes, E. Ferrett, Introduction to Health and Safety in Construction: For the NEBOSH National Certificate in Construction Health and Safety, Routledge, 2015 (2015) New Yourk, United States.
8. Y. Wang, X. Dai, C. Bailey, *J. Constr. Steel Res.,* 67 (2011) 1149.
9. B. Gardelle, S. Duquesne, V. Rerat, S. Bourbigot, *Polym. Adv. Technol.*, 24 (2013) 62.
10. F. Ahmad, S. Ullah, M.S. Hamizol, *J. Appl. Sci.,* 12 (2012) 2631.
11. W. Xing, P. Zhang, L. Song, X. Wang, Y. Hu, *Mater. Res. Bull.*, 49 (2014) 1.
12. R. G. Puri, A.S. Khanna, *Prog. Org. Coat.*, 92 (2016) 8.
13. Z. Liu, M. Dai, Y. Zhang, X. Gao, Q. Zhang, *Prog. Org. Coat.*, 95 (2016) 100.
14. S. Ullah, F. Ahmad, *Sol. ST. Phen.*, 185 (2012) 90.
15. S. Ullah, Faiz Ahmad, P.S.M. Megat-Yusoff, N.H.b. Azmi, *J. Appl. Sci.*, 11 (2011) 1630.
16. J. Kaur, F. Ahmad, S. Ullah, P.S.M.M. Yusoff, R. Ahmad, *Arab. J. Sci. Eng.*, 42 (2017) 2043.
17. U. Schulz, P. Trubiroha, U. Schernau, H. Baumgart, *Prog. Org. Coat.*, 40 (2000) 151.
18. S. Ullah, F. Ahmad, A.M. Shariff, M.R. Raza, P.J. Masset, *J. Anal. Appl. Pyrolysis,* 124 (2017) 149.
19. R. Norvaisiene, A. Burlingis, V. Stankevicius, *Mater. Sci-Medzg.*, 16 (2010) 80.
20. M. Jimenez, S. Bellayer, B. Revel, S. Duquesne, S. Bourbigot, *Ind. Eng. Chem. Res.*, 52 (2013) 729.
21. NACE Standard, Joint Surface Preparation Standard, (2000)., in.
22. A. Kinloch, Adhesion and adhesives: science and technology, Springer Science & Business Media, (2012) London, United Kingdom.
23. T.H. Nguyen, N. Eshtiaghi, K.P. Hapgood, W. Shen, *Powder Technol.*, 201 (2010) 306.
24. ASTM B117-16, Standard Practice for Operating Salt Spray (Fog) Apparatus, ASTM International (2016), West Conshohocken, PA, www.astm.org, in.
25. B. Jegdić, J. Bajat, J. Popić, S. Stevanović, V. Mišković-Stanković, *Corros. Sci.*, 53 (2011) 2872.
26. S. Ullah, F. Ahmad, *Polym. Degrad. Stab.*, 103 (2014) 49.
27. S. Ullah, M.A. Bustam, A.M. Shariff, G. Gonfa, M. Ayoub, M. Raihan, *Int. J. Electrochem. Sci.*, 12 (2017) 1642.
28. Z.Ž. Lazarević, V.B. Mišković-Stanković, Z. Kačarević-Popović, D.M. Dražić, *Corros. Sci.*, 47 (2005) 823.
29. J.B. Bajat, I. Milošev, Ž. Jovanović, V.B. Mišković-Stanković, *Appl. Surf. Sci.,* 256 (2010) 3508.
30. S. Kakooei, M.C. Ismail, B.A. Wahjoedi, *Int. J. Electrochem. Sci.*, 8 (2013) 3290.
31. S. Palraj, M. Selvaraj, S. Muthukrishnan, Effect of pretreatments on electrodeposited epoxy coatings for electronic industries, *AIMS Mater. Sci*., 3 (2016) 214.
32. Y. Zuo, R. Pang, W. Li, J. Xiong, Y. Tang, *Corros. Sci.*, 50 (2008) 3322.
33. W. Chen, R.-G. Du, C.-Q. Ye, Y.-F. Zhu, C.-J. Lin, *Electrochim. Acta*, 55 (2010) 5677.
34. B. Jiang, Y. Ge, Corrosion prevention of magnesium alloys (2013), Elsevier Inc. Chapters.