



THESIS

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**COMPOSITE ZINC-FLY ASH COATING
ON
MILD STEEL**

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**BACHELOR OF CHEMICAL ENGINEERING
UNIVERSITI MALAYSIA PAHANG**

**COMPOSITE ZINC-FLY ASH COATING
ON
MILD STEEL**

EZREE FARHAN BIN ABU HANIPAH

Thesis submitted in partial fulfilment of the requirements
for the award of the degree of
Bachelor of Chemical Engineering

**Faculty of Chemical & Natural Resources Engineering
UNIVERSITI MALAYSIA PAHANG**

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Dedicated to my supervisor and student master

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ABSTRACT

The corrosion was one of the most crucial issues which mankind has been faced. Corrosion naturally impacts our daily life through chemical reactions that occur on the surface of metal and will impair the function of metal. In addition, the production of waste fly ash from a thermal power plant shows an increasing each year and been disposed at the landfill. One of the solutions to prevent corrosion occurs on metal by coated the surface of metal. Recently, fly ash found that have a potential to work as an anti-corrosion agent because of the composition of fly ash rich in metals such as iron (Fe), aluminium (Al), silicon (Si) and magnesium (Mg). The objectives of this paper were to study the corrosion behaviour and to study the physical properties of anti-corrosion zinc –fly ash coating. Therefore, Zinc-fly ash coatings were deposited on mild steel substrates with used sodium silicate as a binder. Zinc powder and fly ash powder was being sieved first in order to get the fine powder for composite coating and mix with sodium silicate. The surface of mild steel has been coated with zinc-fly ash coating. The physical properties of zinc-fly ash coating were being tested by an adhesion test by the cross-cut method. The corrosion behaviour of coating were being tested by immersion test in 3.5% sodium chloride (NaCl) solution for 30 days and potentiodynamic polarization curve in 3.5% sodium chloride solution. The results from adhesion test found that by the addition of fly ash in composite coating increased the adhesion strength of the coating than without fly ash. The result from immersion test concluded that the addition of fly ash had less rested on the surface of mild steel. The result of potentiodynamic polarization curve show the zinc-fly ash coating exhibits a more protective film than a pure composite zinc coating with 50% compared to pure zinc coating.

ABSTRAK

Pengaratian adalah salah satu isu yang sering dihadapi dan berlaku secara semulajadi. Pengaratian boleh memberi impak kepada kehidupan kita melalui tindak balas kimia yg berlaku di permukaan logam dan akan menjejaskan fungsi logam. Di samping itu, pengeluaran sisa abu terbang dari kilang kuasa haba menunjukkan peningkatan setiap tahun dan telah dilupuskan di tapak pelupusan. Salah satu penyelesaian untuk mencegah logam berkarat dengan melapiskan cat dipermukaan logam. Baru baru ini, abu terbang didapati mempunyai potensi untuk berfungsi sebagai agen anti karat kerana komposisi abu terbang yang kaya dengan logam seperti besi (Fe), aluminium (Al), silikon (Si) dan magnesium (Mg). Objektif kajian inin adalah mengkaji tingkah laku pengaratian dan mengkaji sifat fizikal zink-abu terbang cat. Oleh itu, zink-abu terbang didepositkan pada substrat keluli dengan natrium silikat yang digunakan sebagai pengikat. Serbuk zink dan serbuk abu terbang ditapis untuk mendapatkan serbuk yang halus untuk komposit cat dan dicampurkan dengan natrium silikat dan permukaan keluli telah disalut dengan cat zink-abu terbang. Ciri-ciri fizikal zink-abu terbang cat telah diuji dengan ujian lekatan. Ciri-ciri pengaratian diuji dengan ujian rendaman dalam larutan 3.5% natrium klorida selama 30 hari dan pengiraan polarisasi potentiodynamik dalam 3.5% larutan natrium klorida. Keputusan dari ujian lekatan mendapatin bahawa dengan menambah abu terbang dalam komposit cat akan meningkatkan kekuatan lekatan cat berbanding tanpa abu terbang. Hasil daripada ujian rendaman mendapati bahawa penambahan abu terbang akan mengurangkan pengaratian terhasil. Hasil keputusan ujian pengiraan polarisasi potentiodynamik menunjukkan zink-abu terbang cat mendapati terbukti mengurangkan pengaratian sebanyak 50% berbanding dengan cat tanpa abu terbang.

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LIST OF ABBREVIATIONS

Al ₂ O ₃	Aluminum Oxide
CaO	Calcium Oxide
Cd	Cadmium
Cr	Chromium
Cu	Copper
Fe ₂ O ₃	Ferromagnetic
Hg	Mercury
MgO	Magnesium Oxide
Mn	Manganese
NO ₂	Nitrogen Dioxide
Pb	Lead
SiO ₂	Silicon Dioxide
SO ₂	Sulfur Dioxide
SO ₃	Sulfur Trioxide
Sr	Strontium
Th	Thorium
U	Uranium
XRF	X-ray fluorescence

CHAPTER 1 INTRODUCTION

INTRODUCTION

1.0 Background of the study

1.1.1 What is Corrosion

Corrosion may be determined as a physicochemical interaction between a metal and its environment that results in changes in the characteristic of the metal. Consequently, corrosion may cause to significant impairment of the function of the metal, the environment, or the technical system of which these elements form a part. Corrosion is the result of an electrochemical reaction that requires an electrolyte solution and a metallic conductor between two separate areas with different potentials i.e., an anode and a cathode. The formation of ferrous oxides (rust) is a well-known effect of the corrosion process when iron and steel corrode, but many other metals are also subject to corrosion. The green-colored patina on copper and the white rust on zinc are other examples of corrosion products (Sørensen & Kiil, 2009).

The driving force in the corrosion of metals, illustrated in Fig. 3 for steel, is the potential difference between the anodic and cathodic sites. The overall potential difference between the anode and cathode is associated with a potential determined by the tendency of the involved half cells to occur spontaneously i.e., the standard potential of a half-cell reaction (Sørensen & Kiil, 2009).

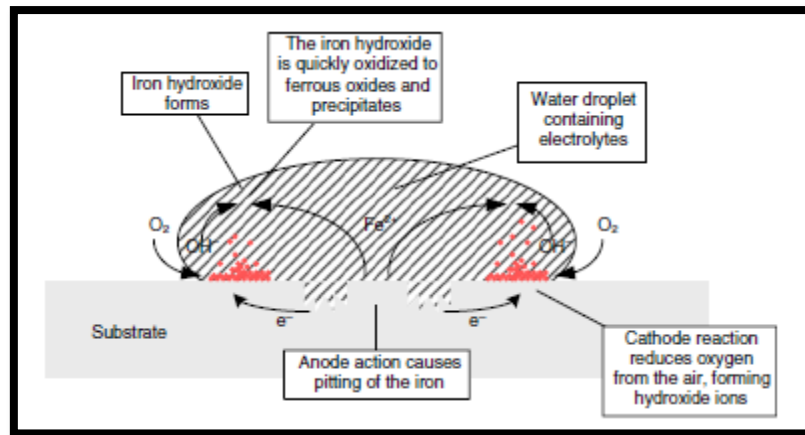
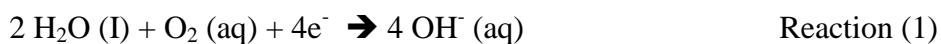


Figure 1: Illustration of the corrosion process of steel in the presence of water, oxygen, and electrolytes. Pitting refers to a form of extremely localized corrosion that leads to the creation of small holes in the metal (Sørensen & Kiil, 2009)

1.1.2 Mechanism of Corrosion

On a steel surface, some areas are anodic while other areas are cathodic. At the cathode, oxygen is reduced on a catalytically active surface of oxidized metal, mainly to hydroxyl ions as given in reaction (1), but other reaction products such as peroxides, superoxides, and radicals may also be formed. At the anode, several corrosion reactions take place, and the net result is the production of ferrous ions and electrons given in reaction (2) (Sørensen & Kiil, 2009).



1.1.3 What is Fly-Ash

Fly ash, generated in thermal power plants, is recognized as an environmental pollutant. Since wide scale coal firing for power generation began in the 1920s, many millions of tons of ash and related by-products have been produced. Annual production of coal ash worldwide is estimated to be around 600 million tonnes, with fly ash constituting about 500 million tonnes at 75–80% of the total ash produced. Fly ash is generally gray in color, abrasive, mostly alkaline, and refractory in nature.

1.2 Motivation

Fly-ash was a waste that been generated in thermal power plant and the amount of waste fly ash will be increasing every year and mostly fly ash has disposed as landfill. Disposal of waste fly ash to the environment will soon be too costly if not forbidden.

The used of fly-ash as an additive in coating pigment found that has led to increasing the effectiveness of anti-corrosive behaviour of coating because of the chemical composition in fly-ash. Therefore, some of the research proves that the other utilization of waste fly ash can be used for additive for making of coating because fly-ash have potential react as the anti-corrosive and can improve the properties of materials.

1.3 Problem Statement

The corrosion is one of the most crucial issues which mankind has been faced. Corrosion naturally impacts our daily life trough chemical reactions that occur between metal or metal alloys and their environment because metals turn to return to their stable, oxidized state. Corrosion occurs with both industrial, domestic environment and the corrosion of metal surface increases significantly as the structure ages. Corrosion should be prevented by the safest and lowest cost method during the earliest stage of corrosion through the use of coating on the surface of metal. The application of zinc metal particles for corrosion protection has been examined for more than 50 years. According to several studies, zinc rich paints (ZRP) are well known as efficient organic coatings to protect metallic substrates from corrosion.

In recent years, it has been shown that by addition amount of fly-ash powder in pigment coating are able to increase the physical and effectiveness of anti-corrosion coating (Pan, Georgiou, & Gavras, 2008). Fly-ash has tremendous potential in corrosion protection purposes because of fly-ash being rich in metal oxides like SiO_2 , Al_2O_3 , Fe_2O_3 and TiO_2 (Ruhi, Bhandari, & Dhawan, 2015).

1.4 Research Objective

The following are the objectives of this research:

1. To study the corrosion behaviour of anti-corrosive zinc-fly ash coating.
2. To study the physical properties of zinc-fly ash coating.

1.5 Scope of Study

The following are scopes of this research:

1. Preparation of the pigment coating zinc-fly ash in sodium silicate solution as binder with different ratio of fly-ash used.
2. Cross-cut tape will used to study the quality of adhesion strength of coating.
3. In immersion test using 3.5% sodium chloride solution, open to air for 30 days.
4. Study corrosion behaviour by measure the potentiodynamic polarization curve.

CHAPTER 2

LITERATURE REVIEW

2.1 Anticorrosive coatings

An anticorrosive coating system usually consists of multiple layers of different coatings with different characteristics and reasons. Depending on the desired properties of the coating system, the individual coats can be metallic, inorganic, or organic. A typical anticorrosive system for highly corrosive marine environments usually consists of a primer, one or several intermediate coats, and a topcoat. The purpose of the primer is to protect the substrate from corrosion and ensure good adhesion to the substrate. For this reason, metallic zinc or inhibitive pigments are often formulated into coatings applied as primers for structures situated in the splash zone or in an atmospheric environment. In addition to adequate resistance to alternating weathering conditions and impacts from objects, the topcoat should also have a high resistance to ultraviolet radiation. The environmental degradation caused by moisture, temperature, and ultraviolet radiation will reduce the lifetime of the coating (Sørensen & Kiil, 2009).

2.2 Protective mechanisms

Anticorrosive coatings are generally classified in accordance with the mechanisms by which they protect a metal against corrosion. Figure 2.1 illustrates the three basic protective mechanisms of anticorrosive coatings: barrier protection, passivation of the substrate surface (inhibitive effect), and sacrificial protection (galvanic effect).

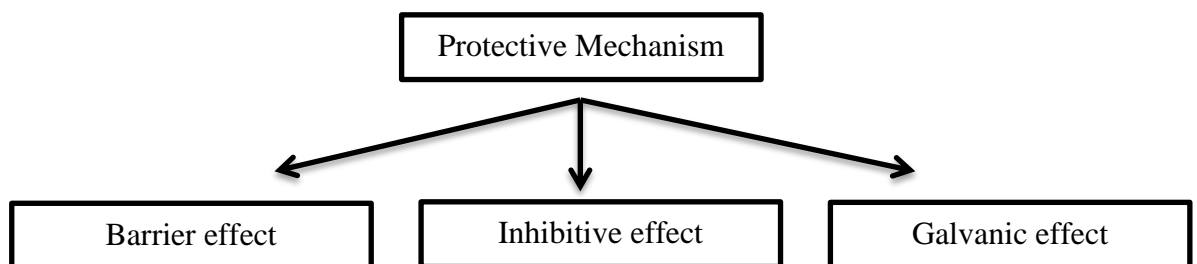


Figure 2.1: Protective mechanisms of anticorrosive coatings (Sørensen & Kiil, 2009)

2.2.1 Barrier Coating Protective

Barrier coatings may be used as primer, intermediate, or topcoat, and are often applied on immersed structures. The barrier coating system is highly dependent on the thickness of the coating system as well as the generic type and nature of the binder system. The delamination of both defect-free and artificially effect barrier coatings has been reported to be significantly decreased when the thickness of the coating is increased (Sørensen & Kiil, 2009).

2.2.2 Inhibitive coatings

Inhibitive coatings are primarily applied as primers because they are solely effective if dissolved constituents can react with the metal. These coatings are mainly applied to substrates subject to environments with a risk of atmospheric corrosion, in particular industrial environments, and are generally not recommended for immersion in water or burial in soil. The anticorrosive mechanism of inhibitive coatings relies on passivation of the substrate and build-up of a protective layer consisting of insoluble metallic complexes, which impede transport of aggressive species by acting as a barrier (Sørensen & Kiil, 2009).

2.2.3 Galvanic effect (Sacrificial coatings)

Sacrificial coatings rely on the principle of galvanic corrosion for the protection of metals against corrosion. This means that the substrate is protected by a metal or alloy that is electrochemically more active than the material to be protected. In this respect, coatings formulated with metallic zinc powder have been extensively employed for corrosion protection of steel structures for several decades. Unlike barrier coatings, sacrificial coatings are only applied as primers because they are only effective if the coating is in direct contact with the substrate due to the requirement of electrical contact between the substrate and the sacrificial metal (Sørensen & Kiil, 2009).

2.3 What is Fly Ash

The Fly ash is produced during combustion of coal in the thermal power plants and generate large amount of bottom ash and fly ash. The quality of the coal ash depends on the properties of coal, combustion efficiency, pulverized coal feed and proper quality control in maintaining the particle size. Larger amount of fly ash as by-products of combustion are generate in India and the quality is very poor. The amount of fly ash increasing because of the demand of the coal as fuel in thermal power plants increase in time (Singh, Kumar, Singh, & Mohapatra, 2016). In India, productions of by-product fly ash presently over 112 million tonnes by thermal power plants. Fly ash quality depends on coal, coal particle fineness, percentage of ash in coal, combustion technique used, air/fuel ratio, burners used, and type of boiler (Dhadse, Kumari, & Bhagia, 2008). Besides, in China large amounts of fly ash are produced factories and thermal power plants and the amount of fly ash increasing by-year. In China about of 480 million tonnes of fly ash were produced in 2010 and the estimated it is about 570 million tonnes by the end of 2015. The quantity of fly ash from thermal plants is massive (Wei, Wang, Ying, Luo, & Ninomiya, 2015).

2.1.1 Characteristic of Fly Ash

Physical properties of Fly ash that have been production at Indian coal combustion it's particle are usually very fine, light weight with the density is 1.97-2.89 g/cc, and spherical with specific surface area, 4000-10,000 cm²/g and it diameter 1-150 micro-meter, refractory and have pozzolaniac ability. Fly ash has dielectric property (dielectric constant, 104) and can be used in electronic application (Dhadse et al., 2008).

The effective treatment of fly-ash has become an attractive environment problem that needs immediate settlement. Despite available technologies, only a small percentage of fly-ash had been commercial application and the rest being buried in landfills (Wang, Han, & Zhang, 2016). Safe disposal of the ash without adversely

affecting the environment and the large storage area required are major concerns. Hence, attempts are being made to utilize the ash rather than dump it. The coal ash is utilized in bulk only in geotechnical engineering applications such as construction of embankments, fills, landfill liners (Salunkhe & Mandal, 2014).

2.1.2 Fly Ash Effect to Environment and Human Healthy.

Fly ash are consider as hazard to environment and Life because of contain trace amount of toxic metal (U, Th, Cr, Pb, Hg, Cd etc) which have negative effect on human health and on plants. Several studies have been carried out to assess hazards caused by Fly ash on environment and plants. The composition SO₂ and NO₂ released from thermal power plant cause acid rain, which corrodes structural surfaces and may affect agriculture by causing yellowing of green leaves. Thermal pollution due to disposal in surface water sources disrupts aquatic life, whereas toxic metals leached contaminate underground water resources. Light and continuous prolonged inhalation causes pneumonitis, allergy, asthma, lung fibrosis, bronchitis, cancer and silicosis. Limited studies have done and potential of silicosis and lung cancer associated with crystalline silica component of fly ash (Dhadse et al., 2008).

Fly ash becomes a serious environmental problem due to their leaching characteristics. During the hydraulic conveying and disposal, coal ash comes with contact with water. The tracing elements present in coal ash migrate to ground water, surface water and soil over a period of time. Therefore, it is necessary to predict the leaching characteristics of the fly ash of thermal power plant to prevent the environmental effects. The tracing elements like manganese (Mn), magnesium (Mg), chromium (Cr), copper (Cu), nickel (Ni), lead (Pb), iron (Fe), zinc (Zn) and cobalt (Co) were determined by ASTM method.

The result shows that the tracing element of Mn, Mg, Cr, and Cu presence in major concentration while element Pb, Fe, Zn and Co are present in minor concentrations. The element Mn shows the maximum leach ability whereas Fe shows the minimum leach ability. These element have effects that can cause harmful effect on human health like heart attack, damage of nervous systems, lung tumor and liver failure etc. (Singh, G., Kumar, S., Singh, M. K., & Mohapatra, S. K., 2016). Fly ash also one of the factors that contribute in air pollution relative to dust emission via wind erosion and a waste product (Panki Katra Fly Ash Contamination).

2.1.3 Fly Ash Anti-Corrosive Properties.

Fly-ash has tremendous potential in corrosion protection purposes because of fly-ash being rich in metal oxides like SiO₂, Al₂O₃, Fe₂O₃ and TiO₂. Further, the fineness in size of fly-ash will improved the pore refinement and minimizes the access to deteriorating agents even at accelerated corrosion process (Ruhi et al., 2015). Research founded that, coated fly ash with copper by electrolysis coatings using Sn–Pd catalyst, in order to impart electrical conductivity to it. In addition, prepared a series of aluminium and nickel coated fly ash using pressure infiltration technique. Recently, a prepared of zinc-fly ash composite coatings on mild steel in order to improve it wear and corrosion resistance (Yeole, Kadam, & Mhaske, 2014).

2.1.4 Fly-Ash used to increase the Physical Properties

The cement industry used fly ash as a raw material for production of concrete because of its pozzolanic properties. There are essentially three applications for fly ash in cement, including (1) replacement of cement in Portland cement concrete, (2) pozzolanic material in the production of pozzolanic cements, and (3) set retardant ingredient with cement as a replacement of gypsum. As pozzolan greatly improves the strength and durability of concrete, use of ash is a key factor in their preservation. Use of fly ash as a partial replacement for Portland cement is generally limited to Class F fly ash. Due to the spherical shape of fly ash particles it can also increase workability

of cement while reducing water demand. Unlike typical soils used for embankment construction, fly ash has a large uniformity coefficient consisting of silt-sized particles (Ahmaruzzaman, 2009)

The increase of surface hardness of metallic materials leads to an increase in their adhesion abrasive resistance. This observation is in full agreement with the results given here, as it was reported earlier that the fly ash particles increase potentiate the hardness of the zinc coating (Pan, Georgiou, & Gavras, 2008)

2.2 Zinc Pigment Coating

Metal particles are frequently incorporated into coatings on metallic substrates for various purposes. Zinc metal particles have been widely used to improve the corrosion resistance of coatings for more than 50 years. There also have been many studies on zinc-rich organic coatings in order to improve their corrosion resistance (Park, Yun, Kim, Song, & Park, 2012). For heavy duty industries and industrials condition like offshore and under marine, zinc rich coating have been used to protect steel substrates because of its galvanic protection ability and work as protection barrier. Many researchers have begun to pay attention to zinc rich coating due to its anticorrosion ability. The performance of the coatings was largely affected by the pigment and binder ratio (Park et al., 2012). Anticorrosion pigments make an important contribution to the performance of protective coatings. In other words, they are capable of enhancing the protection through chemical/electrochemical or physical mechanisms (Nederi et al, 2012).

In zinc-rich primers, zinc is used to produce an anodically active coating. Zinc will behave as an anode and sacrifice itself to protect the metal, which becomes a cathode. The resistance towards corrosion is dependent on the transfer of galvanic current by the zinc primer, but as long as the conductivity in the system is preserved, and as long as there is sufficient zinc to act as anode, the metal will be galvanically protected. The electrochemical activity in a damaged zinc coating system results in

the formation of zinc corrosion products, which tend to seal the pores between the zinc particles to a point at which the system becomes electrically nonconductive, as illustrated in Fig. 3. Subsequent protection is attributable to the barrier effect by corrosion products (Sørensen & Kiil, 2009).

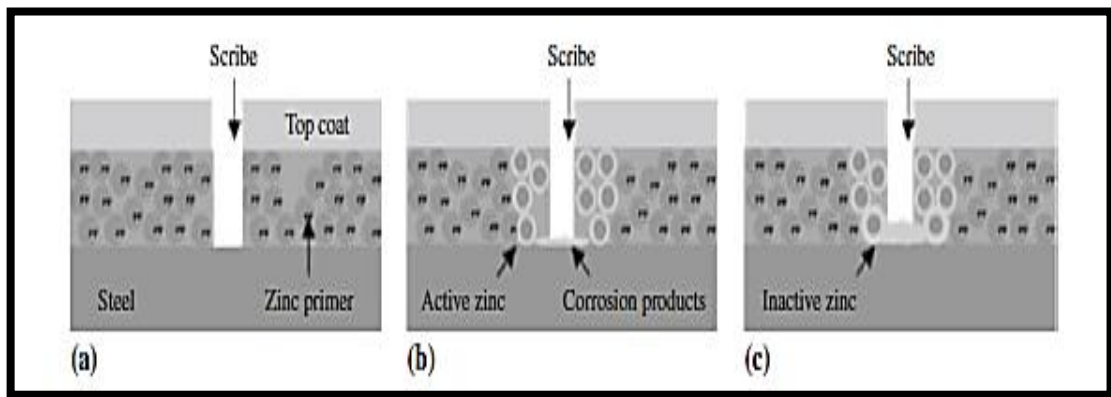


Figure 2.2: Stylistic sketch of the working mechanisms of a zinc-rich coating system. (Step a) Damage is introduced down to the steel surface. (Step b) Zinc around the score is still active while zinc-iron corrosion products are being built up. (Step c) The zinc is not galvanically active any longer but the coating provides barrier and inhibitive protection (Sørensen & Kiil, 2009)

2.2 Technique Used for Coating

The size of the powder of coating and fly ash needed to sieve to 180 μ m in order to obtain a fine powder. Further, the fineness in size of fly-ash will improve the pore refinement and minimizes the access to deteriorating agents even in the accelerated corrosion process (Chen, Li, Fu, & Zhou, 2012). The steel specimens were obtained by cutting the steel to a dimension of 10 mm x 40 mm x 2 mm for corrosion studies. The specimens were polished by grinding them with emery papers of 120, 600 and 800 grit size to attain the smooth finish and to remove the oxide layer on the surface of the mild steel. Thereafter, the specimens were degreased with acetone and then dried (Pan, Georgiou, & Gavras, 2008).

The paint was formulated by addition of fly-ash as additive with different of percent by weight of the solution of sodium silicate (Na_2SiO_3). Sodium silicate (Na_2SiO_3) solution was prepared with water solvent and then the binder solution was allowed to rest for 2 hours until the entire compound dissolved at room temperature (Chen, G et al., 2012). The amount of pigment (zinc powder + fly-ash) fixed to be 30 percent by mass (wt %) and amount of binder fixed to be 70 percent by mass (wt %) of sodium silicate solution. The different amount of fly-ash was used for each formulation of coating. The table 1 shows the amount of fly-ash used for each formulation of coating. Then, the pigment and binder solution were mixed until getting a harmonized blend of coating. Finally, the prepared coating was applied to the mild steel and dried at room temperature for a while and the coating was heated in the oven at 100°C for 2 hours (Chen et al., 2012). The total weight for the each sample in this experiment is 10 g.

2.3 Binder

The binder has been used of this primer is inorganic silicate. In contrast to organic binders in normal paints, this composition provides dry heat resistance up to $400\text{-}450^\circ\text{C}$ (ZINC SILICATE (INORGANIC) - Tambour Paint, 2009). Sodium and potassium silicates in aqueous solutions have physical and chemical properties that are useful in bonding and coating applications. When applied as a thin layer on or between surfaces of other materials, the silicate solution dries to form a tough, tightly adhering inorganic bond or film which can exhibit the following characteristics, low cost, non-flammable, resistant to temperature up to 3000°F , bondable to metals and strong and rigid (Bulletin 12-31 Bonding And Coating Applications of PQ). The use of sodium silicate as a binder has considerably increased in recent years. Sodium silicate was used for moulding because it enables the preparation of the mould and cores without any need for drying and baking in certain cases even without ramming the sand (Rabbii, 2001).

Sodium silicate (Na_2SiO_3) was selected as a binder for coating because sodium silicate can meet certain criteria for binder, including moderate ability to suspend powders and generally produce medium viscosity coating, low softening temperature and high wetting ability to mild steel and cost effectiveness and applicability. Glass ceramic coating with excellent chemical inertness, self-healing ability and high temperature stability are very promising material for the protection of mild steel against oxidation. Glass ceramic coatings mixed with a water based binder liquid can offer significant protection for mild steel after additional heat treatment. During the heat treatment, the coatings materials fuse to form a strong adherent coating otherwise cracks appear easily. Sodium silicate (Na_2SiO_3) solution was prepared with water solvent and then the binder solution was allowed to rest for 2 hours until the entire compound dissolved. The coating was heated at 100°C for 2 hours until it transformed to gel state completely. The advantages of sodium silicate as a binder after mixed with coating powder, the powder filled in the voids and the binder molecules presented an effective cross linking to the powders. The application of coating is suitable in high temperature when sodium silicate used as a binder (Chen et al., 2012).

2.4 Characterization and Property Measurements

2.4.1 X-ray fluorescence (XRF) characterization

The chemical composition of fly ash particles was found by X-ray Florescence Technique. XRF analysis of fly-ash was carried out by X-ray fluorescence S8 Tiger. Fly-ash was being sieved into $180\ \mu\text{m}$ before analysed in order to get the fine particles of pigment of coating (Pan, Georgiou, & Gavras, 2008).

2.4.2 Potentiodynamic Polarization Measurement

For the investigation of the corrosive behaviour of the produced zinc and zinc-fly ash coated mild steel specimens, potentiodynamic polarization measurements were conducted in a 3.5% sodium chloride (NaCl) solution (pH = 5.5) at 20 °C temperature with the aid of an EG&G Potentiostat–Galvanostat Instrument. The electrochemical experimental set-up was composed of a three electrode cell using a platinum grid as counter electrode and saturated silver-silver chloride electrode Ag/AgCl as reference, the coated samples connected to the working electrode (Helmi Azizul & Noda Kazuhiko, 2007). The scan rate was always 0.2 m V/ s. (Pan, Georgiou, & Gavras, 2008).

2.4.3 Immersion Test in 3.5% Sodium Chloride Solution

The anticorrosion properties of coating were also evaluated by immersion test in 3.5% sodium chloride (NaCl) solution for a period of 30 days. There has four samples with different percent by weight of fly-ash that work as additive to coating pigment that been tested. After 30 days, the performance of the coating against corrosion was examined visually (Pan, Georgiou, & Gavras, 2008.).

2.4.3 Adhesion Strength Testing

The adhesion test on coated mild steel was examined according to ASTM D3359 standard. The results are described according to the percentage of area removed during the test (5B for no removed area, 4B for 5% removed area, 3B for 5% to 15% removed area, 2B for 15% to 35% removed area, 1B for 35% to 65% removed area and 0B for greater than 65% removed area). The adhesion test results were compared with those of pure zinc coated mild steel. This observation could be attributed to the fact that the embedded hard fly ash particles hinder the abrasive movement of the indenter in the composite coating (Panagopoulos, C., Georgiou, E., & Gavras, A., 2000).

As a conclusion, zinc coating was effective in protecting steel against corrosion. The principle of this protective action is attributed to the fact that zinc being higher than iron in the electromotive series of the elements reacts first in any environment conducive to the ionic dissolution (oxidation) of metals, thereby protecting the steel substrate. Then, by using fly-ash as an additive into coating pigment will increase the physical and electrochemical of coating. The reasons were in fly-ash they have several of chemical composition such as iron (Fe), silicon (Si), aluminium (Al) and calcium (CA). In addition, sodium silicate was used as a binder for process of coating because of sodium silicate can meet certain criteria for binder, including moderate ability to suspend powders and generally produce medium viscosity coating, low softening temperature and high wetting ability to mild steel and cost effectiveness and applicability. Sodium silicate known as glass ceramic, with excellent chemical inertness, self-healing ability and high temperature stability are very promising material for the protection of mild steel against oxidation. There were three common methods for coating analysis, immersion analysis, and adhesion analysis and measure potentiodynamic polarization curve.

CHAPTER 3

MATERIALS AND METHODS

3.0 Introduction

The chemical used in this project is to evaluate the effectiveness of paint coating based on physical appearances and corrosion behaviour was zinc powder, fly-ash, sodium chloride, acetone and sodium silicate. Sodium chloride and acetone were available in Lab FKKSA UMP. There were three methods to identify the effectiveness of fly-ash as anti-corrosive additive in zinc pigment coating by mechanical testing including adhesion test and immersion test. Lastly, the sample was analysed by potentiodynamic polarization measurement to determine corrosion rate of the coating.

3.1 Material

Sodium silicate (Na_2SiO_3) was purchased from R & M Chemical. Specification chemical for sodium silicate were pH 5% of solution ($\leq 11.5\%$), density (20/40) between 1.350 -1.380, copper (Cu) ($< 0.005\%$), lead (Pb) ($< 0.005\%$) and nickel (Ni) ($< 0.005\%$). Meanwhile, fly ash was obtained from Gimah Plant (Negeri Sembilan, Malaysia). Zinc powder, acetone and sodium chloride were obtained in lab.

Table 3.1: Material Used For Enhanced the Zinc-Fly Ash Anti-corrosive Coating

Material	Description
Mild steel length with dimension 10 mm x 40 mm x 2 mm	For the immersion test, potentiodynamic polarization and adhesion testing.
Acetone Solution	Cleaning solution for clean the surface mild steel before been coated.
Deionised water	Rinsing the mild steel
3.5 % Sodium Chloride solution	For test the immersion solution and potentiodynamic polarization testing.
Sodium Silicate in liquid form	Binder for coating.
Zinc powder and Fly ash	For enhance of anti-corrosive coating.
100, 200 and 800 grit silicon carbide paper (SiC)	Polish on the mild steel surface.
Epoxy Hardener	Used for polarization measurement

3.2 Apparatus

Table 3.2: Apparatus Used For Enhanced the Zinc-Fly Ash Anti-corrosive Coating

Apparatus	Description
Sieve	For sieve the Fly ash
Oven	For dried the coating on the mild steel
EG&G Potentiostat– Galvanostat Instrument	For corrosion behaviour
Brush	For coated the coating on mild steel surface
Knife	Adhesion testing



Figure 3.1: Epoxy and Hardener, Slow setting epoxy adhesive

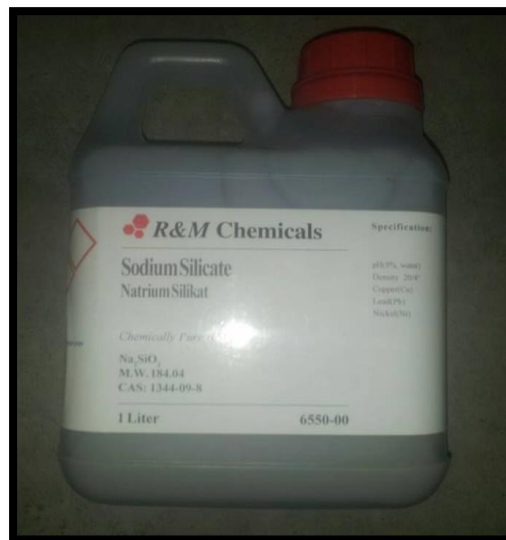


Figure 3.2: Sodium Silicate in Liquid form was purchased from R & M Chemical



Figure 3.3: Fly-ash Powder

3.3 Methodology

3.3.1 Flow Chart Process

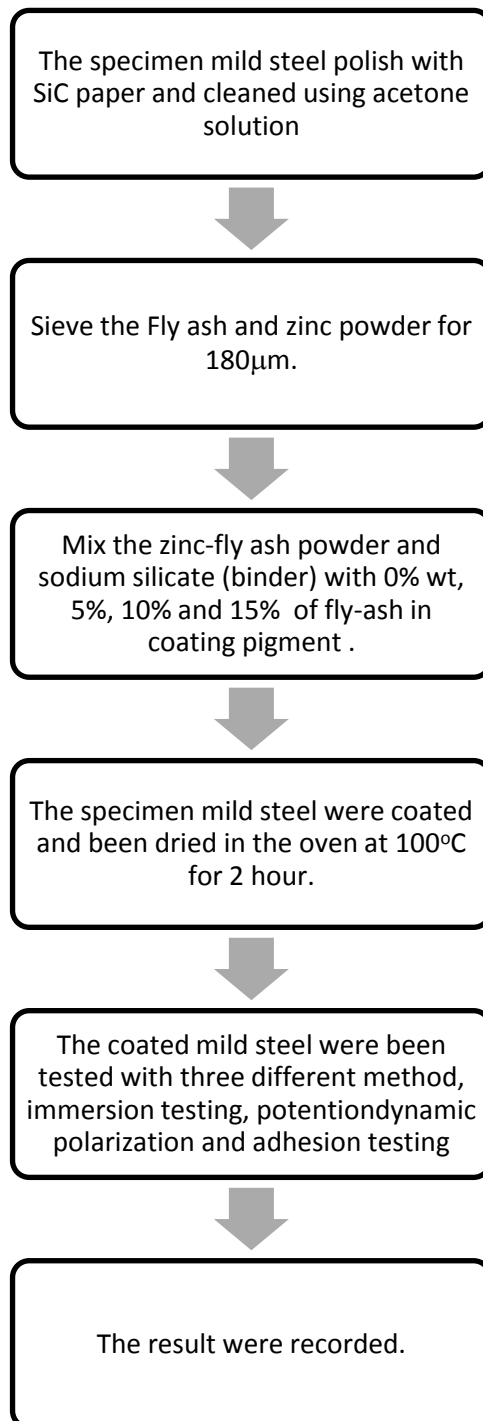


Figure 3.4: Overall flow of procedure of anti-corrosion coating zinc-fly ash

3.4 The Specimen Mild Steel Polish With SiC Paper and Cleaned Using Cleaning Agents

The steel specimens were obtained by cutting the steel to a dimension of 10 mm x 40 mm x 2 mm for corrosion studies. The specimens were polished by grinding them with emery papers of 120, 600 and 800 grit size to attain the smooth finish and to remove the oxide layer on the surface of the mild steel. Thereafter, the specimens were degreased with acetone and then dried (Pan C. N. et al., 2008). After the pickling of mild steel, the surface was cleaned with deionised water to remove surface contaminants (Panagopoulos, C., Georgiou, E., & Gavras, A., 2009).



Figure 3.5: Mild steel before been polish with SiC paper



Figure 3.6: Mild steel after been polish with SiC paper (100, 200 and 800 grit silicon carbide paper)

3.5 Sieving the Fly ash

The size of the powder of coating and fly ash needed to sieve to $180\mu\text{m}$ in order to obtain a fine powder. Further, the fineness in size of fly-ash will improve the pore refinement and minimizes the access to deteriorating agents even in the accelerated corrosion process (Chen et al., 2012). Filter the fly ash in order to get the average particle with filter first before runs the experiment because fly ash that get are not purely, due to impurity in the fly ash. The reason for fly ash been filter because to remove any lumps and granules.

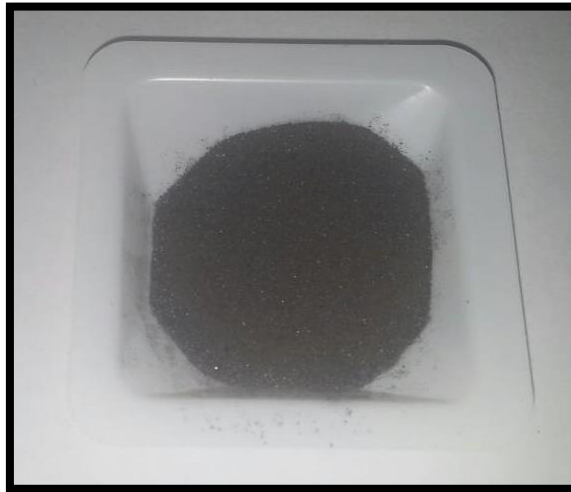


Figure 3.7: Fly-ash was been sieve to $180\mu\text{m}$



Figure 3.8: Zinc Powder

3.6 Mixing Process of Zinc - Fly Ash Powder and Sodium Silicate

The paint was formulated by addition of fly-ash as additive with different of percent by weight of the solution of sodium silicate (Na_2SiO_3). Sodium silicate (Na_2SiO_3) solution was prepared with water solvent and then the binder solution was allowed to rest for 2 hours until the entire compound dissolved at room temperature (Chen, G et al., 2012). The amount of pigment (zinc powder + fly-ash) fixed to be 30 percent by mass (wt %) and amount of binder fixed to be 70 percent by mass (wt %) of sodium silicate solution. The different amount of fly-ash was used for each formulation of coating. The table 3.3 shows the amount of fly-ash used for each formulation of coating. Then, the pigment and binder solution were mixed until getting a harmonized blend of coating.

Table 3.3: The Composition of Fly-ash used as additive

No. of Sample	Binder (70 wt% of Sodium Silicate) g	Pigment 30 wt%	
		Zinc Powder (g)	Fly-Ash (g)
A0	7 g	3.00 g (100%)	0.00 g (0%)
A1	7 g	2.85 g (95%)	0.15 g (5%)
A2	7 g	2.70 g (90%)	0.30 g (10%)
A3	7 g	2.55 g (85%)	0.45 g (15%)



Figure 3.9: The mix process of zinc powder, fly-ash and binder

3.7 The Specimen Mild Steel were Coated and Dried in the Oven

The composited coating was applied on the mild steel and dried at room temperature for a while and the coated mild steel was heated in the oven at 100°C for 2 hours (Chen, G et al., 2012).

3.8 Testing Conducted on Coating

3.8.1 Potentiodynamic Polarization Measurement

For the investigation of the corrosive behaviour of the produced zinc and zinc-fly ash coated mild steel specimens, potentiodynamic polarization measurements were conducted in a 3.5% sodium chloride (NaCl) solution (pH = 5.5) at 20 °C temperature with the aid of an EG&G Potentiostat–Galvanostat Instrument. The electrochemical experimental set-up was composed of a three electrode cell using a platinum grid as counter electrode and saturated silver-silver chloride electrode Ag/AgCl as reference, the coated samples connected to the working electrode. The scan rate was always 0.2 m V/ s. (Pan C. N. et al., 2008). Before that, the uncoated surface must seal with epoxy hardeners to make sure that the uncoated surface affect the measurement.

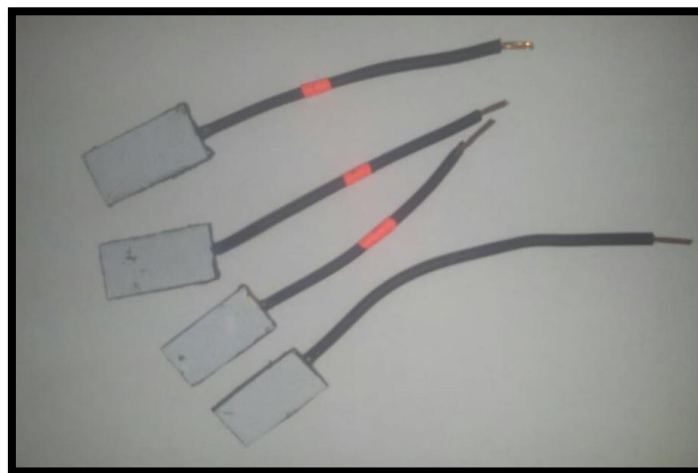


Figure 3.10: Sample for potentiodynamic polarization measurement



Figure 3.11: The uncoated surface of mild steel was been seal with epoxy hardeners

3.8.2 Immersion Test in 3.5% Sodium Chloride Solution

The anticorrosion properties of coating were also evaluated by immersion test in 3.5% sodium chloride (NaCl) solution for a period of 30 days. There has four samples with different percent by weight of fly-ash that work as additive to coating pigment that been tested. After 30 days, the performance of the coating against corrosion was examined visually.

3.8.3 Adhesion Testing

The adhesion test on coated mild steel was examined according to ASTM D3359 standard. The results are described according to the percentage of area removed during the test (5B for no removed area, 4B for 5% removed area, 3B for 5% to 15% removed area, 2B for 15% to 35% removed area, 1B for 35% to 65% removed area and 0B for greater than 65% removed area). The adhesion test results were compared with those of pure zinc coated mild steel. This observation could be attributed to the fact that the embedded hard fly ash particles hinder the abrasive movement of the indenter in the composite coating (Panagopoulos, C., Georgiou, E., & Gavras, A., 2000).

The following procedure was used to carry out adhesion test on the surface of coating.

Materials: knife, cellophane tape

Technique: Cut the carbon coated steel by a knife with uniform pressure and draw cutting edge and cross the original cut at 90° to make a lattice pattern. Brush in diagonal for five times time each, tape over the cut and remove with tape.

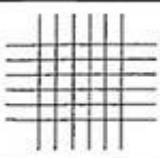
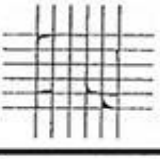


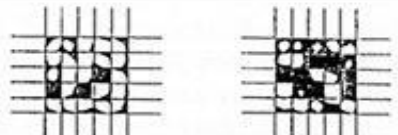
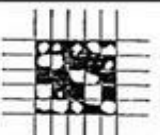
Classification	% of Area Removed	Surface of Cross-cut Area From Which Flaking has Occured for 6 Parrallel Cuts & Adhesion range by %
5B	0% None	
4B	Less than 5%	
3B	5 - 15%	
2B	15 - 35%	
1B	35 - 65%	
0B	Greater than 65%	

Figure 3.12: Standard ASTM D 3359 for Adhesion Testing

Description for Classification coating:-

- ASTM CLASS 5B – “edges cut are completely smoothly, none of the square lattice is detached.”
- ASTM CLASS 4B – “small flakes of the coating at the intersection of cuts. Not significantly greater than 5 % is affected.”
- ASTM CLASS 3B – “Coating has flakes along edge and intersection. Significantly greater than 5 % but not significantly greater than 15 %.”
- ASTM CLASS 2B – “Coating has flakes along edges of cuts, fully in a different part of squares. Significantly 15 % but not greater than 35 %.”
- ASTM CLASS 1B – “Coating has flaked along edges of cuts, some squares have detached fully and significant.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

This chapter present a results and discussion of each testing that been analysed on coating zinc fly-ash on mild steel compared to a pure zinc coating.

4.1.1 Determine the best ratio between pigment and binder

Before the experiment started, the ratio of pigment of coating and binder (sodium silicate) was determined by using trial and error method. The table below shows the trial and error method for ration of pigment of coating and binder (sodium silicate).

Table 4.1: The different ratio between pigment coating and binder

Sample	Ratio Pigment of Coating	Ratio of Sodium Silicate (binder)	Result	Time for coating to dried, hour (h)
1	4	6	Too dried, difficult to coated on mild steel	4-7
2	3	7	Smooth Coating	10-15
3	2	8	Smooth Coating	15-20
4	1	9	Too dilute coating	>24

The best ratio between pigment and binder was 3:7 because the surface of the coating was very smooth and dried faster than the other ratio.

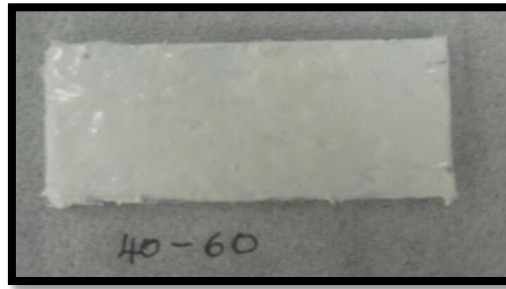


Figure 4.1: Ratio of coating between pigment and binder was 4:6

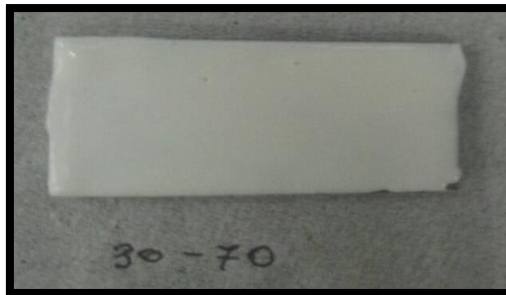


Figure 4.2: Ratio of coating between pigment and binder was 3:7

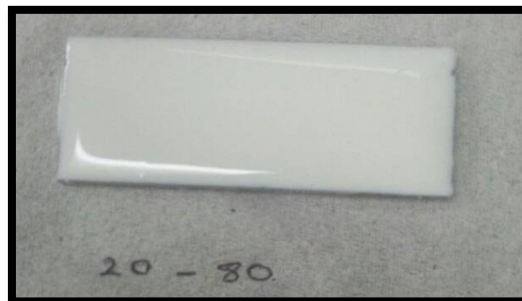


Figure 4.3: Ratio of coating between pigment and binder was 2:8

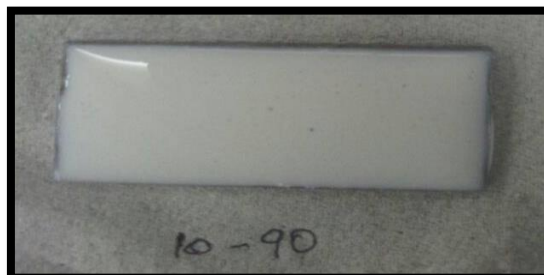


Figure 4.4: Ratio of coating between pigment and binder 1:9

4.2 X-ray fluorescence (XRF) characterization

The table 4.2 show the composition of fly-ash particles from XRF analysis after been sieved for 180 micrometre. Further, the fitness in size of fly-ash will improved the pore refinement and minimizes the access to deteriorating agents even at accelerated corrosion process (Ruhi et al., 2015). The table 4.2 below shows the composition in.

Table 4.2: The composition of fly-ash particles analysed by XRF technique

Composition	Concentration (%)
Iron(Fe)	12.12
Silicon (Si)	11.87
Aluminium (Al)	3.43
Calcium (Ca)	1.43
Potassium (K)	0.65
Titanium (Ti)	0.58
Sulphur (S)	0.41
Zirconium (Zr)	0.32
Phosphorus (P)	0.27
Strontium (Sr)	0.23
Manganese (Mn)	0.16
Barium (Ba)	0.09
Chromium (Cr)	0.08
Magnesium (Mg)	0.08

The result shows that the composition in the fly-ash has a lot of different types of chemical substance. It found that fly-ash being rich in metal such as iron (12.12 %), silicon (11.87 %), aluminium (3.43 %) and titanium (0.58 %). Then, fly-ash has tremendous potential in corrosion protection because of rich in metal (Ruhi et al., 2015). Based on the result from XRF analysis, fly-ash found that has many chemical substances that had potential to increase the adhesion strength of coating. The reason that fly-ash can increase the adhesion strength of the coating because of in composition of fly-ash it contain many types of metallic metal iron (Fe), aluminium (Al), silicon (Si) and titanium (Ti) (Pan C. N. et al., 2008).

4.2 Immersion Test

The anticorrosion performance of the zinc fly-ash coating was evaluated by the immersion test in 3.5% sodium chloride (NaCl) solution. The zinc fly-ash pigments in the coating with three different ratio of fly-ash (5%, 10% and 15%) were compared with the pure zinc pigment coating. Figure 1 shows the surface images of the coated steel exposed to the solution of sodium chloride. The corrosion resistance of coated steel was improved with the increase quantity of fly-ash used as additive, the result shows in figure 1. For composited pure zinc coating had most rust on mild steel surface compared to the sample that had used fly-ash as additive. Based on the figure 1-(B), it show that the used of 5 wt % of fly-ash had similar in amount of rust on the surface of mild steel. For samples that used 10 wt% of fly-ash and 15 wt% of fly-ash in pigment coating, it shown improvement in anti-corrosive behaviour of coating on mild steel surface compared to the pure zinc coating.

The order of anticorrosion efficiency of the coated specimen roughly agreed with the corrosion resistance of the incorporated particle itself, as shown in the previous particle immersion test. This fact demonstrated that the improvement of the corrosion resistance of fly-ash positively affected the anticorrosion performance of the coating (Navarchian, A. H et al., 2015).

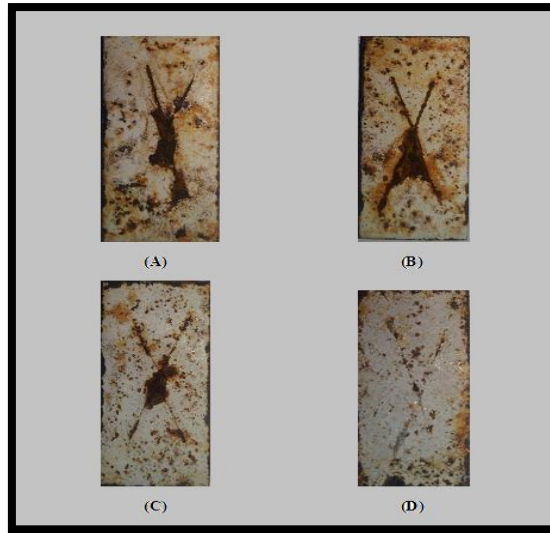


Figure 4.5: Mild Steel test coated with (A) 0 wt% of fly-ash, (B) 5 wt% of fly-ash, (C) 10 wt% of fly-ash and (D) 15% of fly-ash immersed in NaCl (3.5 wt.%) solution for 30 days.

4.3 Adhesion Test

Figure 4.6 shows the result of adhesion testing on surface of the coating area on mild steel. By comparing the surface of zinc fly-ash coating obtained from the scratch testing experiments with pure zinc coating, it could be said that the addition of fly ash particles in the zinc powder coating had higher grade and adhesion values. This observation could be attributed to the fact that the embedded hard fly ash particles hinder the abrasive movement of the indenter in the composite coating.

The increase of surface hardness of metallic materials leads to an increase in their adhesion abrasive resistance. This observation is in full agreement with the results given here, as it was reported earlier that the fly ash particles increase potentiate the hardness of the zinc coating (Pan C. N. et al., 2008). The table 4.3 below shows the result from adhesion test. It was found that the adhesion strength between zinc-fly ash composite coating and the mild steel substrate higher compared to pure zinc coating to mild steel.

Fly ash is dependent on the concentration of the deposit in which it is deposited. Due to the deposition, the mechanical and electrochemical properties of the coating were increased. However, excessive use of surfactant leads to deposit brittleness and reduce in cathode area (Jeyaraj, Sudhambarish, Karthik, & Arulshri, 2015). The composite coating possesses enhanced properties such as wear, corrosion, oxidation resistance, dispersion hardening or self-lubrication, so that they can protect the metal substrates from severe environments during the applications. That's why the grade for sample A3 was 4B compared to sample A1 and A2 were 5B. So it found that, the suitable percent by weight of fly-ash were between 5%wt to 10 %wt. The result of adhesion testing was shown in table 4.3 below.

Table 4.3: Classification of Coating Specimen with different ratio of Fly-Ash

Sample	Classification of Coating
A0	2B
A1	5B
A2	5B
A3	4B

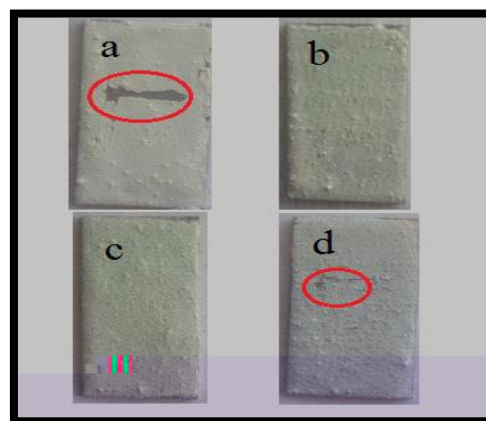


Figure 4.6: Adhesion Testing (a) pure zinc coating, (b) 5% of fly-ash, (c) 10% of fly-ash and (d) 15% of fly-ash

4.4 Potentiodynamic Polarization Measurements

For polarization measurement, sample A3 (15% of fly-ash) was selected because of have less corrosion after immersion test and will compared to the pure composite zinc coating on mild steel surface.

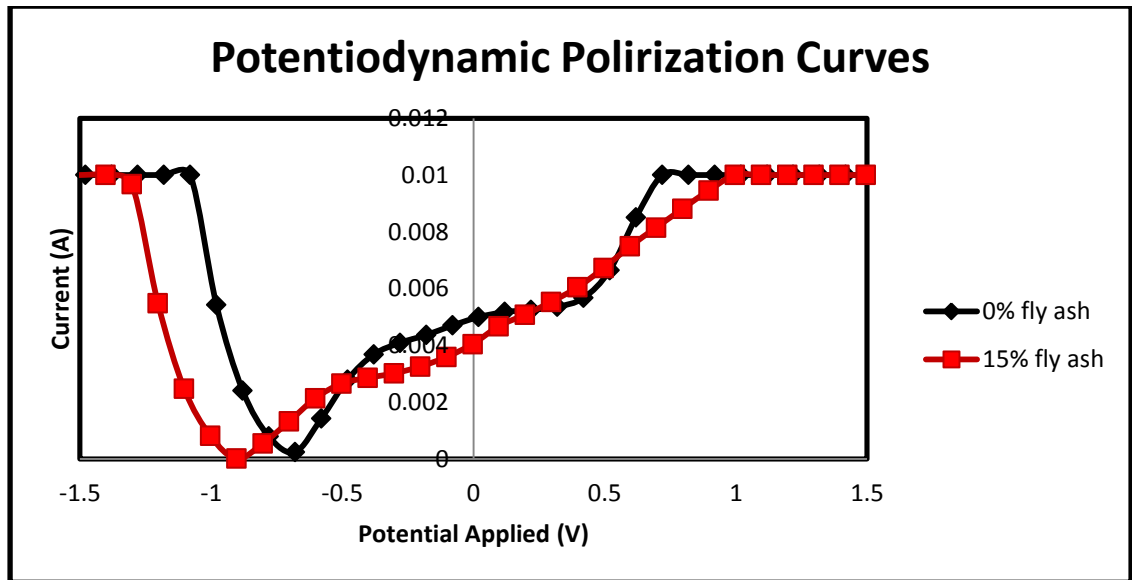


Figure 4.7: Polarization curves of composite zinc-fly ash coating and pure composite zinc coating on mild steel surface.

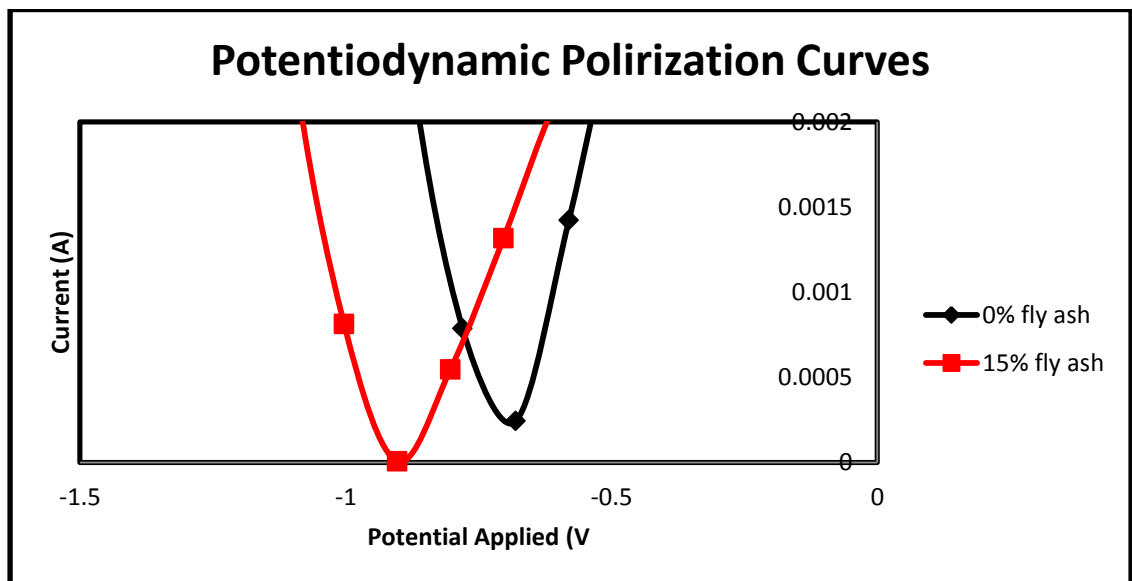


Figure 4.8: Polarization of composite zinc-fly ash coating and pure composite zinc coating on mild steel surface after has been zooming.

The polarization curves obtained from a composite of zinc-fly ash coating and pure composite zinc coating on mild steel surface in 3.5% sodium chloride solution were shown in figure 4.7 and 4.8. The electrochemical parameters (E_{corr} and i_{corr}) for both pure zinc and zinc-fly ash coated mild steel samples were shown in table 4.4. It is proved that, the corrosion potential of composite zinc-fly-ash coating ($E_{corr} = -1.412$ V) is more negative compared to corrosion potential of pure composite zinc coating ($E_{corr} = -1.2546$ V), the zinc-fly ash coating exhibits a more protective film (improved passivation behaviour) than a pure composite zinc coating. This is proven by the lower current densities from onset of passivation through the transpassive region.

Then, by compared the current densities for both samples, it found that current densities for zinc-fly ash coating ($i_{corr} = 214.870 \mu\text{A}/\text{cm}^2$) have lower value than the current densities for pure zinc coating sample ($i_{corr} = 732.650 \mu\text{A}/\text{cm}^2$). Then zinc-fly ash coating has more protection characteristics than pure zinc coating (Youssef, Koch, & Fedkiw, 2004).

From the figure 4.7, the corrosion rate and polarization resistance for both samples has been calculated and shown in table 4.5. From table 4.5 shown corrosion rate for pure zinc coated mild steel (8.5134 mm/year) compared to zinc-fly ash coated mild steel (2.4968 mm/year), the zinc-fly ash coated mild steel is effective compared to pure zinc coated mild steel. Then, for polarization resistance pure zinc coated mild steel (105.970 Ω) compared to zinc-fly ash coated mild steel (214.050 Ω), the zinc-fly ash coated mild steel more effective because of had a high in polarization resistance (Youssef et al., 2004).

Table 4.4: Electrochemical Parameters of Sample A0 (pure zinc coated mild steel) and Sample A3 (zinc-fly ash coated mild steel) in 3.5% Sodium Chloride Solution

Sample	Electrochemical Corrosion Potential (E_{corr}), (V)	Current Density (I_{corr}), ($\mu\text{A}/\text{cm}^2$)
A0	-1.2546	732.6500
A3	-1.4121	214.8700

Table 4.5: Corrosion rate and Polarization Resistance of Sample of A0 (pure zinc coated mild steel) and Sample A3 (zinc-fly ash coated mild steel) in 3.5% Sodium Chloride Solution

Sample	Corrosion Rate (mm/year)	Polarization Resistance (Ω)
A0	8.5134	105.970
A3	2.4968	214.050

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The physical and corrosion behaviour composite of zinc-fly ash coating were studied by three different method, adhesion test, immersion test and potentiodynamic polarization analysed. Based on the results, shown that by added of fly-ash powder in composite zinc coating increased the adhesion strength of coating with surface of mild steel compared to pure composite zinc coating. It proved that, by addition of quantity of fly-ash can increased the adhesion strength of coating because of the increased of surface hardness of metallic materials in chemical composition of fly-as leads to an increase in their adhesion abrasive resistance. For immersion analysis, the result shows that the composite zinc-fly ash coating improved the anti-corrosion behaviour compared to the pure composite zinc coating in 3.5% sodium chloride solution. Then, the result of potentiodynamic polarization test shown that the corrosion potential of composite zinc-fly-ash coating was more negative (-1.4121 V) compared to the corrosion potential of a composite pure composite zinc coating (-1.2546 V), the zinc-fly ash coating exhibits a more protective film (improved passivation behaviour) than a pure composite zinc coating. In conclusion, the addition of fly-ash particles was found to increase the adhesion strength of the coating and zinc-fly ash coating was observed more anti-corrosive than a pure zinc coating with 50%.

5.2 Recommendation

The application work is limited by using small sample plate only for laboratory research. For more advanced, recommend using a large plate for each sample instead of small plate for immersed in the sea water. Furthermore, for the future work need to test the thickness of the coating and the method to apply coating on surface of mild steel.

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