Jurnal Akademika Kimia, 11(1): 19-25, February 2022
ISSN (online) 2477-5185 | ISSN (print) 2302-6030
http://jurnal.fkip.untad.ac.id/index.php/jak/

Analysis Redox Reaction on Zinc-Coating Thickness Test in Metal Processing Industry of Small and Medium Enterprises

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Received 27 September 2021, Revised 17 January 2022, Accepted 10 February 2022
doi: 10.22487/j24775185.2022.v11.i1.19-25

Abstract

This study discusses the implementation of redox reactions on the thickness test of the galvanized (zinc-coated) layer using the JIS-H-0401 standard to help Small and Medium Enterprises (SMEs) industries. Some of the finished products go through the galvanizing process in the metal processing industry. Quality constraints, especially related to the thickness of the galvanic (zinc-coated) layer, have become an object that is less controlled because of the limitations of the test equipment used. This research uses an experimental method applied to one of its products: a flat washer with SPCC material and a thickness of about 2.20 mm. SPCC - SD material is classified as low carbon steel based on its carbon content. 5-unit flat washers were identified with sample codes A, B, C, D, and E. Flat washers have an internal diameter of 22.65 - 22.7 mm. In comparison, the outer diameter is between 43.75 - 43.80. The redox reaction process used HCl (hydrochloric acid) with a concentration of 1 M, which was diluted with water (H_2O). The zinc thickness test results showed that the flat washer has an average thickness of 10.52 microns with a minimum and maximum thickness variation of 10.66 -10.72 microns.

Keywords: Flat washer, hydrochloric acid, low carbon steel redox reactions, small and medium enterprises, zinc-coated

Introduction

Fasteners are crucial to the mechanical and structural integrity of assembled components and systems in fixed and mobile infrastructures. (Chung et al., 2019). Zinc-coated parts are among the most widely used materials in marine platforms, automobiles, port docks, the construction industry, and concrete structures due to their lower cost and relatively higher anti-corrosion performance (Liu et al., 2017; Jin et al., 2018). To improve the service life of the fasteners and economic consideration, sacrificial coatings are normally finished with conversion coatings such as hexavalent chromium, trivalent chromium, and non-chromium passivation. (ASTM International 2016).

The corrosion rate depends on external and internal factors (Karthick et al., 2020). The process for checking the thickness of the galvanized in medium-small and micro-businesses (MSMEs) has still become an issue. MSMEs have limitations in providing product quality testing instruments due to limiting sources of budgets. Availability of product quality test equipment, especially those related to the zinc/galvanized plating process, is often neglected. As a result, the quality of the products produced by MSMEs is not controlled to be less competitive. Most MSMEs rely only on vendors' ability, so it is not easy to maintain the quality of the products. The redox reaction uses 1.0 M of the hydrochloric acid solution, diluted by water and expected to help SMEs with galvanized products. The results of the mass coating test will be converted to the thickness of the galvanic/zinc layer, namely by utilizing the surface area of the workpiece and the density of the zinc layer used.

MSMEs are vital in achieving the Sustainable Development Goals (SDGs) for nations. MSMEs are a pillar of a nation's economy. According to Act of the Republic of Indonesia Number 20 the year 2008, the definition of MSMEs is productive economic activities that stand alone (UU No. 20 Tahun 2008). In Indonesia, MSMEs can absorb up to 97 percent of energy and contribute to the Gross Domestic Product (GDP) of up to 57 percent (Faqir, 2021). In addition, MSMEs also contributed 14.17 percent of total exports and 58.18 percent of total investment in Indonesia (Putri, 2019). Unlike in developed countries, the SME sector in Indonesia is not integrated with large industries. It seems to have a world of its own. This middle-class entrepreneur is not connected to the big companies

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that are supposed to be his market. In 2017, there were 62.9 million MSMEs, and in 2018, there were 64.2 million MSMEs. There were more than 65 million MSMEs in Indonesia in early 2021. (Christy, 2021). According to this data, only about 18 to 19% of MSMEs are already linked with large corporations. This condition makes it difficult for MSMEs to improve product quality; however, a low-cost and straightforward method is required.

Research on using hydrochloric acid has been conducted, including corrosion testing (Geethamani et al., 2019), synthesis processes (Charoenchokpanich et al., 2021), and zinc layer thickness testing (Ashadi et al., 2002). Some of the literature relevant to this research, who researched the corrosion rate of galvanized metal (coated with zinc) using the chemical solution of hydrochloric acid. This study was conducted using an experimental method by performing three variations of hydrochloric acid concentration 0.5, 1, and 2 M as input variables. The output variable in this study is the corrosion rate measured in mL/second. The fixed variables in the study were defined as 50 mL of hydrochloric acid volume and 3 grams of zinc mass. The reaction rate was measured by calculating the H_2 gas per unit time. The results showed that the test with a high concentration of hydrochloric acid had a faster reaction rate (Ashadi et al., 2002).

A study using zinc and hydrochloric acid was also conducted (Baik & Fray, 2001). The redox reaction research was carried out in the electrowinning process of zinc from a solution of zinc chloride activated by hydrochloric acid and carried out in a cation exchange membrane cell. Current efficiency is correlated with sediment morphology. The sediment had more minor surface defects, which acts as an active site for hydrogen adsorption, shows higher current efficiency. The preferred texture (110) is the observation of a precipitate growing in a highly acidic solution containing gelatine. High temperature (40 °C) and high current density decrease current efficiency and preferred texture. The high current efficiency of electrolysis in 2.0 M of hydrochloric acid and 0.1 M $ZnCl_2$ has sufficient quantities for the zinc concentrate washing process.

Further research involving zinc and hydrochloric acid compounds was also carried out (Saleh & Khalil, 2007). The study was aimed at the corrosion behaviour of zinc in 0.1 M Hydrochloric acid solution containing various concentrations of ampicillin in the range ($2 \times 10^{-4} - 1 \times 10^{-3}$) M. Corrosion rate was measured using zinc weight loss measurements and polarization curves. The results showed that the corrosion rate of zinc increased with increasing temperature from 293 to 323 K and the inhibitory efficiency value of ampicillin increased with increasing temperature. The results showed that inhibitor caused a protective efficiency of 88.8% when a concentration of 0.001 M was used at 323 K. The corrosion rate in the presence and absence of acid solution can calculate the coverage (θ) of metal surfaces. The value (C/θ) and the corresponding concentration of ampicillin (C) were linear relationships, indicating that the inhibitory action was mediated by adsorption. The performance of this inhibitor can be optimized by utilizing the operating temperature and concentration of the inhibitor. Ampicillin is a suitable inhibitor for zinc corrosion in 0.1M of hydrochloric acid.

In contrast with the previously studied, this study used an experimental method using five flat washers with a total surface area of 4741.6-4759.2 mm^2. The test was conducted using a 1 M hydrochloric acid solution diluted in 53.3 mL H_2O. The dilution was carried out to avoid Fe oxidation in the metal. This study aims to determine the thickness of the zinc coating layer in a galvanized part for helping MSMEs to be able to maintain the quality of the galvanized/plated product at the vendor.

Methods

Material and testing instruments

Safety procedures were fully controlled and should be followed safety requirements during the experimental process. Personal protection equipment such as masks, glasses, and gloves should follow the material safety data sheet is also implemented and strictly controlled. Healthy protocols are also followed by established regulations to prevent the transmission and spread of Covid-19 (Khoirudin et al., 2021). The experiment setup used measuring tools and instruments, including beakers, glass, digital calipers, and scales. The digital caliper and digital scale used have an accuracy level of 0.1 mm and 0.1 gram, respectively. This testing was adequately conducted in the Mechanical Engineering laboratory, Buana Perjuangan University, Karawang. The materials used in this study were 1.0 M of hydrochloric acid and 5-unit flat washer diameter with material thickness, inside and outside dimensions being 2.0, 22.7, and 43.8, respectively. Five unit flat washers were identified with A, B, C, D, and E. **Figure 1** presents five samples of the flat washer to be tested. 5 liter of demineralization water was provided for dilute hydrochloric acid.

Hydrochloric acid is commonly used to neutralize alkaline agents, as a bleaching agent, in the production of chlorides; for refining or producing tin and tantalum; for pickling and cleaning metal products (Verma & Khan, 2016), in electroplating; treatment for removal of scale and dust; acid pickling of steel, and removal zinc coating (Fouda et al., 2019).

The surface area of the flat washer must be measured and calculated to determine the thickness of the zinc layer. The flat washer's surface area was divided into three sections., namely L1, L2, and L3. The segmentation L1, L2, and L3 was the crosssectional area of the flat washer surface presented in **Figure 1**. The outer dimension (OD) and inner dimension (ID) were measured using calipers with an accuracy of 0.01 mm and a maximum length of 200 mm. The following process is to calculate the thickness of the zinc layer, which begins with weighing the flat washer before and after the redox reaction. W1 and W2 data are flat washers considering data before and after the redox reaction.

For basic system neutralization, the hydrochloric acid is used as a laboratory reagent, a catalyst and solvent in organic synthesis, and the production of fertilizers and dyes, in the preparation of various food products, as well as in the photographic, textile, and rubber industries, for hydrolyzing starch and proteins. The physicochemical properties of hydrochloric acid are presented in **Table 1** (Abdollahi & Nikfar, 2014).

Table 1. Physicochemical properties

Parameters	Remarks
Appearance	Colorless
Odor	The pungent odor of hydrogen chloride
Solubility	Infinite in water with slight evolution of heat. Soluble in cold water and diethyl ether
Density	1.18
pH	For HCl solutions: 0.1 (1.0 N), 1.1 (0.1 N), 2.02 (0.01 N)
%Volatiles by volume at 21°C	100
Boiling point	53°C
Melting point	-74°C
Specific gravity	1.1–1.19

The instrumentation used included a digital caliper and a digital scale with an accuracy of 0.1 mm and 0.1 gram, respectively. Before the checking process, personal protective equipment (PPE) was prepared to meet safety and environmental regulations. The coating mass check refers to the JIS G 3313 Annex JF standard (JIS G 3313, 1998). The hydrochloric acid 1.0 M needs diluting with water to meet the JIS-H-0401 standard of 0.3 M concentration (JIS H 0401, 1999).

Diluted of hydrochloric acid-water solutions

A total of 25 mL of 1.0 M of hydrochloric acid was diluted properly using 53 mL of H_2O to meet the JIS-H-0401 standard. Dilution is the process of decreasing the solute concentration, namely the HCl-H_2O solution. The diluting process means adding more solvent without adding more substance as solute. The number of moles, the volume of solvent for decreasing concentration, and mass of 25 mL of hydrochloric acid with a molarity of 1.0 M were calculated using equations (1), (2), and (3) (Chang & Overby, 2008).

$$n = \frac{M}{V} \quad (1)$$

$$M_1V_1 = M_2V_2 \quad (2)$$

$$m = n.Mr \quad (3)$$

where n, M, V, m, and Mr are the moles of a substance (moles), the molarity of the substance (moles/liter or Molars), the volume of the solution (liters), the mass (grams), and the relative mass of the compound (grams/mole), respectively.

Test for zinc coatings

The testing process begins by diluting 1.0 M of hydrochloric acid 25 mL to 0.3 M and 83.3 mL in volume. The coating mass test aims to determine the thickness of the zinc layer on galvanized parts.

The calculation of the coating mass was carried out according to equation (4) (JIS H 0401, 1999)

$$C_m = \frac{w_1 - w_2}{A}.10^6 \quad (4)$$

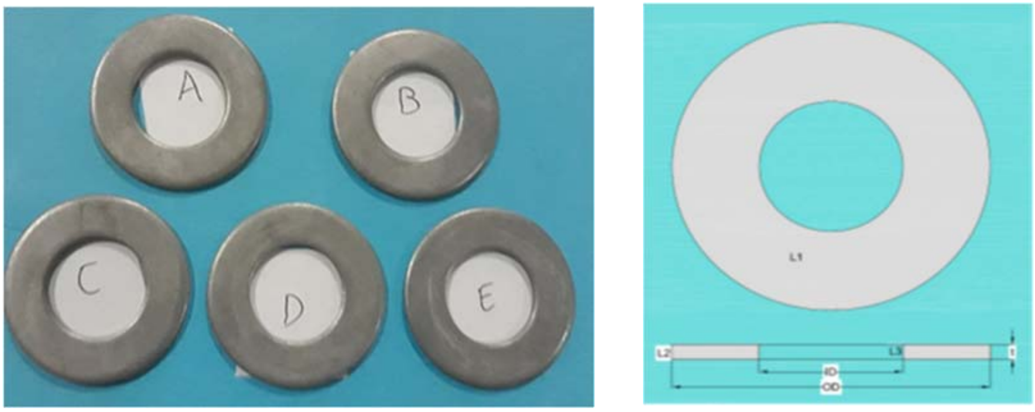
where C_m, w_1, w_2 and A is the coating mass (gram/mm^2), mass before and after a test (in grams), an area in (mm), respectively. The following process calculates the thickness of the coating layer, t_c (zinc thickness), and calculates using equation 5 (Ashadi et al., 2002).

$$t_c = \frac{m}{\rho}.10^6 \quad (5)$$

where m, and ρ, are m mass of zinc (in gram), the zinc density (in gram/ cm^3). The zinc density was determined at about 7.14 g/cm^3 (Thakur & Nandedkar, 2014; Lin et al., 2018). The flat washer dimensions are needed to determine the total surface area. The flat washer dimensions are carried out based on the geometry shown in **Figure 2**.

Redox reaction

Redox reactions are defined by the actual or formal transfer of electrons between chemical species, most commonly with one species (the reducing agent) undergoing oxidation (losing electrons) and another species (the oxidizing agent) undergoing reduction (gains electrons). The chemical reaction that occurs in testing the thickness of the galvanized layer is a redox reaction (reduction and oxidation). A reduction reaction is when electrons gain, and the oxidation number decreases. Oxidation reactions are the opposite of reduction reactions. The number of electrons decreases in the oxidation reaction, and the oxidation number increases (Chang & Overby, 2008). The chemical reactions during the testing process are shown in equation (6), and the balancing chemical reaction is shown in equation (7) (Chang & Overby, 2008).



(a) (b)

Figure 1. Flat washer identification (a) and geometry (b)

$$Zn_{(s)} + HCl_{(aq)} \rightarrow H_{2\,(g)} + ZnCl_{2\,(aq)} \quad (6)$$

$$Zn^0_{(s)} + 2H^I Cl_{(aq)} \rightarrow H^0_{2(g)} + Zn^{II} Cl_2 \quad (7)$$

Zinc and hydrochloric acid are being reduced, and oxidizing agents are. This reaction produces hydrogen gas, considered a clean and efficient energy carrier (Sun et al., 2018). The chemical equations for both reactions are shown in equations (8) and (9), respectively (Chang a& Overby, 2008).

$$Zn^0 - 2\,e^- \rightarrow Zn^{II} \text{ (oxidation reaction)} \quad (8)$$
$$2H^I + 2\,e^- \rightarrow 2H^0 \text{ (reduction reaction)} \quad (9)$$

In this case, zinc functions as a reducing agent due to losing electrons, while hydrogen functions as an oxidant due to receiving the electrons (Saleh et al., 2020). Electron transfer (redox reaction) occurs during the dissolution and deposition of a metal-rich phase (Mathur et al., 2018).

Results and Discussion

Dilution process analysis

The dilution process is required to meet JIS-H-0401 standards. To produce 4 moles of hydrochloric acid can be calculated using equation 7. The JIS G 3313 ANNEX JF standard suggests a concentration of a hydrochloric acid solution of about 0.3 M (JIS G 3313 1998). It is recommended that 25 mL of 1.0 M of hydrochloric acid be diluted. The concentration of 0.3 M of hydrochloric acid in a solution of 4 moles was calculated using equation (8), and the result is as follows.

$$M_1V_1 = M_2V_2,$$

$$1\text{M} \times 25\text{mL} = 0.3\text{ M} \times V_2$$

$$V_2 = \frac{1\text{M} \times 25\text{mL}}{0.3\text{M}} = 83.33\text{ mL}$$

The above calculation adds 58.33 mL of 1.0 M of hydrochloric acid 25 mL of air (83.33-25). The water is weighed using a digital scale of about 58.3 grams.

Analysis of zinc coating thickness

Zinc is a white to bluish metal, shiny and diamagnetic, slightly less dense than iron and has a hexagonal crystalline structure. The metal is hard but malleable at 100-150 °C. Zinc is also capable of conducting electricity. Compared to other metals, zinc has a relatively low melting and boiling point, 420 °C for the melting point and 900 °C for the boiling point. Zinc is a chemical element with the symbol Zn and the atomic number 30. When oxidation is removed, zinc is a slightly brittle metal with a silvery-greyish appearance at room temperature. It is the first member of group 12 of the periodic table (IIB) (Jeong et al., 2017). Zinc electroplating is depositing a thin layer of zinc metal on the surface of another metal, known as the substrate. The zinc coating acts as a physical barrier, protecting the underlying metal surface from rust. (Sukarman et al., 2021). Electroplating is accomplished through the electro-deposition process (Sukarman et al., 2020). A zinc-containing chemical bath is used to soak the fasteners. The zinc metal dissolves when direct current is applied to the anode. The free metal ions reach the cathode and coat the buckle in a thin layer (ASTM International, 2016). The length of time spent in the plating bath, the amount of electric current used, and the chemical composition of the tub contribute to the thickness of the zinc plating.

Figure 2. Test for zinc-coated thickness using hydrochloric acid solutions

Meanwhile, the difference is the mass of the zinc layer attached to the flat washer before peeling off with a redox reaction. The equations (4) and (5) are used to calculate the coating mass and thickness of the zinc thickness coating. Table 1 shows the five flat washers' washer surface area, coating mass, and zinc thickness data.

The lowest coating thickness value of the flat washer tested was 10.66 microns. The highest value was 10.72 microns, as shown in **Table 2**. This zinc layer has an average thickness of 10.52 microns. The zinc layer commonly used for industrial fastener parts has a thickness of 6-8 microns (Speck, 2015). The test data shows that the average value was more excellent than the minimum standard commonly used in the industry. The thickness of 6-8 microns represents the functional coating thickness of the zinc layer of the external threaded thread and the corresponding decrease in the available diameter of the internal threaded thread for the coating thickness. This standard of zinc coating thickness is used for industry. Referring to this data, the zinc coating thickness on the flat washer has exceeded the industry standard. However, because of the function of the flat washer, the excess thickness of about 2.56 microns is not a quality issue but rather a production cost issue due to the time during the electroplating process (Chung et al., 2019). McCune et al. (2016) reported that increasing the treatment duration allows for the deposition of thicker coating layers.

Redox reaction analysis

Saturation occurs when a solution of a substance can no longer dissolve that substance, and additional amounts of that substance appear as a residue in physical chemistry. The redox reaction analysis aims to determine the saturation level of the 0.3 M of HCl 83.33 mL solution. It is hoped that this analysis will allow the HCl solution to be estimated if used to test the thickness of the zinc layer with the same parts and dimensions. The reaction equation in equation (7) can be seen as follows.

$$Zn\,(s) + 2HCl\,(aq) \rightarrow H_2\,(g) + ZnCl_2\,(aq) \quad (10)$$

The relative atomic mass, A_r of Zn, H, and Cl, are 65.4, 1.0, and 35.5 gram/mole, respectively. We get the relative molecular mass, Mr of hydrochloric acid, H_2, and $ZnCl_2$ were 36.5, 2, and 109.9 gram/mole, respectively. The mole and mass of 0.3 M HCl 83.33 mL can be calculated using equations 1 and 2 by the 0.025 moles and 0.9125, respectively. The mole ratio before and after the reaction can be estimated with equation 10. The test results show that the mass of zinc in the flat washer layer is approximately 2 grams or 0.003058104 moles. A saturated solution is a solution with so much solute that it would not dissolve if there were any more. The actual mole ratio of each substance of 4-5 flat washers testing can be estimated using reaction equilibrium 10. This result was calculated and presented in **Table 2**.

Table 2. Flat washer dimension and output variable data's

Flat washer	Dimensions (mm)			Area (mm^2)			Total area		Coating mass	Coating thickness
	OD	ID	T	L_1	L_2	L_3	mm^2	cm^2	g/cm^2	micron
A	43.80	22.70	2.0	2202.9	275.1	142.6	2620.6	26.206	0.0076	10.69
B	43.75	22.70	2.0	2196.1	274.8	142.6	2613.4	26.134	0.0077	10.72
C	43.85	22.70	2.0	2209.8	275.4	142.6	2627.8	26.278	0.0076	10.66
D	43.80	22.75	2.0	2199.4	275.1	142.9	2617.3	26.173	0.0076	10.70
E	43.80	22.65	2.0	2206.5	275.1	142.2	2623.8	26.238	0.0076	10.68

Table 3. Chemical reaction during zinc-coated testing

Process identifications	Chemical reactions balance				
Substance/molecule	Zn	2HCl	→	H_2	$ZnCl_2$
Stoichiometric mole	1	2		1	1
Actual mole	0.0125	0.025		0.0125	0.0125

Saturation occurs when a solution of a substance can no longer dissolve that substance, and additional amounts of that substance appear as a residue in physical chemistry. The equilibrium of the redox reaction between Zn and HCl occurs when 0.0125 mol of Zn reacts with 0.025 moles of HCl. It can be identified visually by the cessation of bubbles from the H_2 gas. H_2 gas and $ZnCl_2$ precipitate formed in this reaction are 0.0125 moles and 0.0125 moles, respectively. In this case, zinc functions as a reducing agent due to losing electrons, while hydrogen functions as an oxidant due to receiving electrons C (Saleh et al., 2020). Electron transfer (redox reaction) occurs during the dissolution and deposition of a metal-rich phase (Mathur et al., 2018).

Conclusions

The redox reaction was successfully conducted to evaluate the zinc thickness testing process with a 1.0 M hydrochloric acid solution diluted to 0.3 M of hydrochloric acid 83.33 mL. The thickness of the zinc layer was measured to be 10.66 -10.72 microns in the tests. Regarding quality, the galvanizing process results produced by SMEs are still below the general standard for component binders. Reaction time will evaluate by using variations of hydrochloric acid concentration as the research response variable in future studies.

Acknowledgment

Authors gratefully to "LPPM Universitas Buana Perjuangan Karawang," that has fully funded this research.

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