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Electrodeposition of Zinc on Iron Plates in the Presence of N,N-Dimethylurea and Sapindus rarak Rind Extract as Additives

Sal Prima Yudha S.[1,*], Akhmad Ues[2], Agus M.H. Putranto[1], Muhamad Alvin Reagen[1], Muhammad Adli Afif[1], Jimmy Askhan[1], Lenon Jus Pentus Tarigan[1]
[1]Department of Chemistry, Faculty of Mathematics and Natural Sciences, University of Bengkulu
WR Supratman, Kandang Limun, Bengkulu City, 38122, Indonesia
[2]CV. Platindo Sinar Metal, Karawang-Bekasi, Indonesia
*E-mail: sp.yudha.s.2022@gmail.com; salprima@unib.ac.id

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Abstract

A study on the addition of natural in the form of Lerak (*Sapindus rarak*) fruit rinds extract (ESR and synthetic additives in the form of N,N-dimethylurea (DMU) compounds in the zinc electrodeposition process on iron plates was carried out at room temperature. The results of this study show that the electrodeposition of zinc in electrolyte solution treated with *Sapindus rarak* extract additive gives a more visually uniform and compact coating morphology based on optical microscopy observations coating product than the electrodeposition product without additive or using N,N-dimethylurea additive. This has been observed using the naked eye and supported by observations under an optical microscope. The existence of zinc coating on the surface of the iron plate is supported by an increase in material thickness (control: 0.051 mm), (DMU: 0.025 mm) and (ESR: 0.028 mm) and an increase in material weight (control: 0.085 g), (DMU: 0.074 g) and (ESR: 0.074 g). In conclusion, the addition of DMU and ESR reduced the thickness and mass gain compared with the additive-free control.

Keywords: N,N-dimethylurea, *Sapindus rarak*, zinc electroplating

1 Introduction

Electrochemical deposition or often called electrodeposition continues to be developed by scientists because this process can place a shiny layer from one metal to another by simply donating electrons to ions in solution [1]. Electrodeposition or often also called electroplating, in the process uses electric current to reduce dissolved metal cations in solution, forming a thin layer on the electrode surface [2,3].

Research related to morphological changes due to additives in the electrodeposition process has been developed quite intensively. As an instant example, extracts from Nicotiana tobaccum leaves have produced morphological changes in the zinc surface coated on mild steel through the electrodeposition process [4]. Cassava leaf extract has also been used as an additive in the zinc deposition process on mild steel and has produced data that the electrodeposition process is very sensitive to changes in additive concentration and electrodeposition time [5].

Another study mentioned that nettle leaf extract (*Urtica dioica* L.) has also been used as an additive in the zinc electrodeposition process in acidic solutions. The results obtained showed that the presence of the extract encouraged the formation of fine grains on the surface of the deposited zinc with an inhomogeneous crystal orientation [6]. Another research study that has been reported is the electrodeposition process using three types of plant extracts, namely ginger, pomegranate and celery extracts. The results showed that ginger extract caused the highest induction of Zn electrodeposition. The results also showed that ginger, pomegranate and celery extracts increased the corrosion resistance of low-carbon mild steel electrodeposited by Zn with a high corrosion rate value compared to uncoated low-carbon mild steel and those undergoing Zn electrodeposition without these plant extracts [7] Henna leaves (*Lawsonia inermis* L.) which have the main content of the dye 2-hydroxy-1,4-naphthoquinone have also been utilized in the zinc electrodeposition process and the results show that the addition of the extract causes an increase in the hardness of the deposit and the inclusion of additives in the deposit [8]. However, the presence of additives does not always result in an effective electrodeposition process. The use of garlic extract in chromium-nickel electrodeposition has reduced corrosion resistance compared to electrodeposition processes that do not use garlic extract as an additive [9].

In addition to plant extracts, several studies have also examined the effect of synthetic chemical compounds as additives in the electrodeposition process. For example, dextrin and thiourea, two commercially available compounds, have been used as additives in the zinc electrodeposition process on mild steel. The results obtained showed the presence of fine, dense and tight crystal grains on the coated metal surface and the product had good corrosion resistance when tested in seawater [10]. Other research results show that DES/H_2O/$ZnSO_4$ (deep eutectic solvent) solvent shows optimal functional performance, based on basic electrochemical data and morphological evaluation [11]. Synthetic compounds, nicotinic acid (NA), boric acid (BA) and benzoquinone (BQ) have also been tested as additives in the electrodeposition process in a DES system composed of choline chloride (ChCl) ethylene glycol (EG) (1:2). The results obtained showed that NA provided shiny zinc electrodeposition results for the first time [12].

Based on the studies that have been reported, the development of research on this path can still be developed to find more economical alternatives and the material is a sustainable resource. The novelty of this study lies in the use of *Sapindus rarak* extract and N,N-dimethylurea as additives in the electrolyte solution for zinc electrodeposition on iron plates. This study is fundamentally based on previous studies related to the use of soapberry rinds extract (*Sapindus rarak*) which has been used as a medium and reducer of silver ions into silver nanoparticles [13] and also as a capping agent that can influence the shape of the cube-shaped sulfur particles from the sodium thiosulfate precursor [14]. In addition, a study has also been conducted on the use of N,N-dimethylurea with citric acid as a precursor for the manufacture of deep eutectic solvent (DES) [15]. Therefore, this study is expected to provide scientific information related to the role of both materials in influencing the course of zinc electrodeposition reactions on iron plates. This scientific information will be useful for development in the industry, especially the zinc electroplating industry, which provides good coating results seen from the brightness of the color and the thickness of the coating. This study explores the use of other materials as alternative additives in the electrolyte solution for zinc electroplating, although it is a preliminary study..

2 Method

2.1 Chemicals and instrumentation

The materials used in this study are demineralized water (aqua-dm), Zinc chloride ($ZnCl_2$), boric acid (H_3BO_3), potassium chloride (KCl). Various types of glassware commonly used in chemical laboratories to support experiments (measuring flasks, beakers, Erlenmeyer flasks, hour glasses) and basic equipment for experiments in the Laboratory such as thermometers, magnetic stirrers, magnetic bars, spatulas, analytical scales. The instruments used in this research are an analytical balance, DC power supply a micrometer screw gauge and an optical microscope.

2.2 Preparation of the material to be coated

The iron plate was cut into several test specimens with a length of 5.0 cm and a width of 2.0 cm. A portion with a length of 1.0 cm was marked at one end for zinc electrodeposition. The test specimens were degreased with a degreasing chemical, namely hexane. The specimens were alternately cleaned for 5 minutes in 35% HCl, rinsed in distilled water, immersed in methanol, air-dried and stored in a desiccator for further experimental processes. After being cleaned with HCl, the iron plate was then etched using 1% H_2SO_4 for 5 minutes.

2.3 Preparation of Extract Sapindus rarak

Fresh *Sapindus rarak* fruits are washed, then the rinds is separated from the pulp. One gram of *Sapindus rarak* rinds is placed in a beaker, to which 50 mL of demineralised water is added. The mixture is stirred for 20 minutes at a temperature of 70°C. Then, once it has cooled slightly, the mixture is filtered using filter paper. This extract is then used as an additive in the zinc electroplating electrolyte solution.

2.4 Preparation of Electrodeposition Solution

The procedure for making the solution follows the method that has been reported by other researchers [10]. The solution for electrodeposition consists of $ZnCl_2$ (71 g/l), KCl (207 g/l) and H_3BO_3 (35 g/l) which is done in the following manner (1 L solution scale): 71 grams of $ZnCl_2$ are weighed, then put into a 1000 mL beaker, followed by 207 grams of KCl and 35 grams of boric acid. After that, add half of the aqua-dm solvent from the beaker, and stir by shaking. After all the solids have dissolved, the aqua-dm is added back up to the 1000 mL beaker boundary mark. The resulting solution is a stock solution that is ready to be used for several electrodeposition conditions designed in this study. Additives in the form of DMU or ESR are added to the main solution.

2.5 Electrodeposition Process

Electrodeposition of zinc on steel was carried out by immersing a portion of the steel specimen and zinc electrode in the plating solution (30 mm depth) in a 250 ml beaker used as a plating bath. The steel specimen was connected to the negative side of the DC supply while the zinc electrode was also connected with a cable to the positive side. Three types of solutions have been used as a medium for the electrodeposition process, namely, a basic electrolyte solution without any additives, as a control solution, then an electrolyte solution containing an additive of soapberry rind extract (*S. rarak*) and then a zinc electrolyte solution that has been given an additive of N,N-dimethylurea each with a concentration of 2% in 250 mL of zinc electrolyte solution. For the *S. rarak* extract, a 2% (v/v) solution was used, whereas for N,N-dimethylurea, a 2% (w/v) solution was used due to differences in form. The electroplating process was carried out for 10 minutes at room temperature (25±2°C). The constant current used in this research is 0.3 A (0.03 A/cm²).

2.6 Pre- and Post-Electrodeposition Material Analysis

Analysis of the surface of the iron plate material to be coated was carried out by observing under a microscope at 3 cm from the iron plate, then measuring the thickness of the iron plate and weighing the iron plate to be coated. Thickness measurements were taken using a screw micrometer at nine different points on the plate, and the average was calculated; weight measurements were taken using an analytical balance.

This data is the pre-deposition data of zinc electrochemically. After treatment, the same procedure was carried out and the data was collected for comparative analysis before and after the electrodeposition process. In addition, a comparison was also analyzed between the control sample (without additives) and the sample that was affected by the presence of both types of additives

3 Result and Discussion

The study began with the preparation of a substrate in the form of an iron plate. The iron plate that has been prepared in this study as shown in Fig. 1. The iron plate that has not received any treatment appears rusty, which is indicated by a yellowish color. Rust is generally known as an oxide form of iron with the general formula $Fe_2O_3.H_2O$ [16]. To minimize the factors beyond control in the zinc electrodeposition process, a sanding process using fine sandpaper is carried out. Sanding is a mechanical process to remove iron oxide compounds (rust) that stick. The color change from yellowish brown to grayish is visible. To ensure that rust has been removed from the surface of the iron plate (substrate), a cleaning process using a hydrochloric acid solution is carried out. The rust removal process using this type of acid produces iron chloride salts that are soluble in water solvents [16].

The process was continued with the stage of opening the pores on the surface of the iron plate using sulfuric acid solution. The three iron plates were then used for the zinc electrodeposition process at a constant current of 0.3A. When electrodeposition is carried out at a constant current, a change in voltage occurs in the electrolyte solution used. In the control solution, the voltage produced was 1.67 V; the ESR additive produced a voltage of 8.05 V; and N,N-dimethylurea produced a voltage of 8.2 V. Synthetic additives like brighteners, levellers, and suppressors, as well as extract additives, adsorb onto the electrode surface and immediately alter the reaction environment.

Based on Fig. 1, there is a similarity in the electrodeposition results. In general, Zn^{2+} ions have been reduced to Zn^0 and form a zinc layer on the surface of the iron plate. However, with the naked eye, it can be seen that the iron plate that has undergone zinc coating in a solution that has been treated with the addition of soapberry rinds extract (*Sapindus rarak*) looks flatter and denser, compared to the coating results using DMU and without the addition of additives (control electrolyte solution). In general, the analysis of the appearance of the zinc electrodeposition process on iron plates was carried out by direct visual inspection, supplemented by an optical microscope at 1600× magnification. This represents a limitation of this study, which is intended as a preliminary investigation to be developed further.

As shown in **Fig. 1**, the results of observations on iron plates under an optical microscope generally show a similar appearance. All three show the presence of rust granules that are yellowish brown in color. Also show a similar appearance on the three iron plates that have undergone sanding treatment, followed by washing using hydrochloric acid and opening the pores using sulfuric acid. All three show the color of the iron plate, grayish. In line with the results of observations using the naked eye as explained, the results obtained from observations using an optical microscope show that the results of the coating using the ESR additive look denser than the results of the coating using the DMU additive or without additives. These results are in line with several previous studies that the use of surfactants can help the electrodeposition process because they contain active compounds that can affect the electrochemical properties of the electrolyte solution and lead to the quality of the resulting metal coating [10].

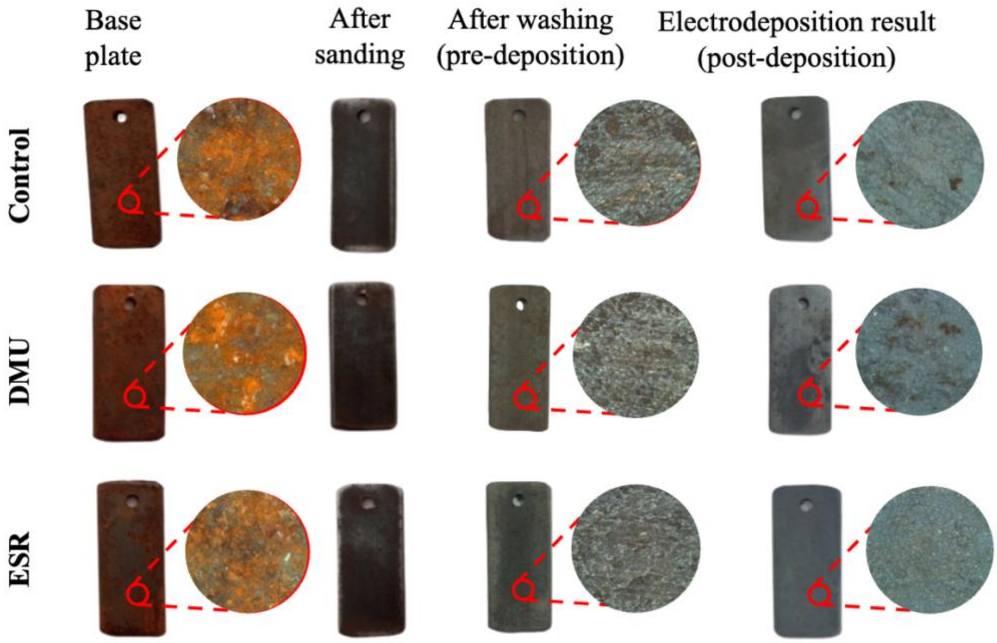

Figure 1. Appearance of iron plates used as substrates for zinc electrodeposition under different treatment conditions, along with magnified surface images observed using an optical microscope at 1600× magnification, as indicated by the circles.

The results of the analysis of changes in the thickness of the pre- and post-deposition iron plates, both using the control solution (without additives) and the addition of both types of additives, can be summarized in **Table 1**.

Table 1. Average thickness changes of iron plates before and after zinc electrodeposition under different additive conditions.*

Condition	Pre-Deposition (mm)	Post-Deposition (mm)	Δ (post-pre) deposition (mm)
Control	2.696	2.747	0.051
DMU	2.760	2.785	0.025
ESR	2.759	2.787	0.028

*The plate thickness results shown are the average of the results of 2 repetitions

Based on **Table 1**, in general, the addition of additives, both DMU and ESR additives, has reduced the thickness of the zinc film on the surface of the coated iron plate, compared to the thickness changes produced in electrodeposition without additives (control). This is predicted because of adsorption of organic additives on the cathodic surface may partially inhibit Zn^{2+} reduction and crystal growth, resulting in lower deposit mass but potentially more uniform coating morphology, but on the other hand increases the level of surface evenness. To strengthen the analysis of the thickness changes, an analysis of the changes in the weight of the iron plate before and after coating was also carried out, both in the control state (without additives) and with the presence of DMU or ESR additives. The results of the analysis of changes in the weight of the pre-deposition and post-deposition iron plates can be summarized in **Table 2**.

Based on **Table 2** and related to **Table 1**, it appears that the addition of iron plate weight after the electrodeposition process shows conformity with the addition of coating thickness. This shows that the amount of zinc coated is indeed greatly influenced by the presence of additives in the form of synthetic compounds and the presence of natural surfactants that have been tested in this study

Table 2. Average mass changes of iron plates before and after zinc electrodeposition under different additive conditions. *

Condition	Pre-Deposition (grams)	Post-Deposition (grams)	Δ (post-pre) deposition (grams)
Control	21.441	21.526	0.085
DMU	22.316	22.39	0.074
ESR	22.312	22.386	0.074

*The plate weight shown is the average of the results of 2 repetitions

According to Faraday's law, the theoretical mass produced during the electroplating process at a constant current of 0.3A over 10 minutes is 0.061 grams. This differs from the mass obtained in the experiment. Because Faraday's Law is perfect and presumes that every electron deposits metal, there is a discrepancy. In actuality, there are physical losses, numerous competing reactions share electrons, and measurement itself increases error.

Current efficiency (CE) was measured by comparing the experimental mass with the theoretical mass, and the results showed that electrodeposition without additives yielded a CE of 139.37%, while ESR and NN DMU yielded a CE of 121.34%. However, when compared with the surface images produced by an optical microscope and via a camera, it can be seen that the dispersion is better with the addition of additives than without them.

In other words, the proton diffusion coefficient decreases due to the presence of *S.rarak* extract in the electrolyte, which may be caused by the specific adsorption of certain molecules contained in the extract onto the active sites on the steel surface. Consequently, the cathodic overpotential increases in the hydrogen evolution reaction [17]. The saponin content in *S. rarak* also plays a role in reducing surface tension, adsorbing onto the cathode surface, and selectively inhibiting crystal growth.

As an additive, dimethylurea-based organic molecules can interact with metal ions (such as Zn^{2+}) to replace some of the water molecules in the solvation shell, thereby limiting the activity of free water through hydrogen bonding interactions. As an additive, these molecules adsorb well onto metal surfaces, forming a stable inorganic and organic protective layer. This enhances the kinetics of metal ions during desolvation and suppresses reactions [18].

4 Conclusion

The addition of *Sapindus rarak* rind extract as a natural additive produced a zinc coating that appeared more visually uniform and compact than those obtained using N,N-dimethylurea or no additive, based on optical microscopy observations. However, the addition of both additives decreased the mass gain and thickness increase compared with the additive-free control, suggesting that the additives may influence Zn nucleation and growth rather than simply increasing the deposition rate. Further characterization, including SEM-EDS, XRD, adhesion testing, and corrosion measurements, is required to confirm the coating composition and evaluate its functional performance.

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