

THE EFFECT OF ADDITION GREEN INHIBITOR D-GALACTOSE ON CORROSION RATE OF ALUMINUM ALLOY 5052 IN SULFURIC ACID (H₂SO₄) MEDIA

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Abstract

Aluminum alloy 5052 (Al5052) is one of metals used as a bipolar plate in a Proton Exchange Membrane Fuel Cell (PEMFC) due to has its light mass and being easy to form and has high conductivity and resistivity properties. This material is prone to corrosion and current knowledge to protect its surface is currently lacking. The product of PEMFC produces electrical energy, hot steam (313–353 K), and water. These conditions have an impact on the degraded bipolar plate caused by the acidic nafion membrane. This increases the risk of corrosion on the cathode side of the bipolar plate. Coating with a green inhibitor using the electrophoretic deposition technique (EPD) is one way to deal with the corrosion that occurs. The analysis method used electrochemical with potentiodynamic polarization techniques, electrochemistry impedance spectroscopy (EIS), Fourier-transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). In this study, green inhibitor D-galactose was used with a concentration of 0.5–1.5 g and an, EPD time of 15–45 minutes in 0.5 M sulfuric acid (H₂SO₄) media pH 1–4. Potentiodynamic polarization analysis at the lowest corrosion current value (I_{corr}) (the inhibitor concentration of 1.5 g with an EPD time of 45 minutes) resulted the corrosion rate of Al5052 before EPD was 0.0075 mmPY, while the corrosion rate of Al5052 after EPD was 0.0041 mmPY (with inhibitors efficiency 45.2%). The FTIR spectrum, broad peak appeared in the range of 3000–3600 cm⁻¹ which referred to the formation of hydrogen bonding of hydroxyl group. Methyl group of D-galactose also appeared on 2918 cm⁻¹ and 2850 cm⁻¹ which attributed to =CH₂ asymmetric stretch and –CH₃ symmetric stretch, respectively. Carbonyl group on 1500–1700 cm⁻¹ represent C=O bond of amide, and aldehyde. Peak 1097–1035 cm⁻¹ which attributed to C-O were connected to the secondary and primary alcohols. The resistance value for Al5052 before and after EPD were 1.2 kΩ/cm² and 2.2 kΩ/cm², respectively. Here we found that the resistance increases with the increasing concentration and time of EPD. The results cross section Al5052 within average 29.8 μm and morphology with SEM Al5052 before EPD showed pitting corrosion. On the other hand, the image of Al5052 inhibitor coating 1.5 g with EPD of 45 minutes showed a smooth surface and visible black lumps, suggesting Al5052 was successfully reduced a corrosion rate by the D-galactose. Our simple and robust method inferred a protection route towards a viable and physically stable green inhibitors.

Keywords: Aluminum Alloy 5052, Electrophoretic Deposition, Electrochemical, D-galactose, Fuel Cell

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1. Introduction

Aluminum alloy 5052 (Al5052) is pure aluminum alloyed with elements Mg 2.2–2.8%, Cu 0.1%, Mg 0.1%, Cr 0.15%, Zn 0.1%, Si and Fe 0.45%. The advantages of Al5052 include high conductivity, ease of formability, and a light mass of 65% steel. Based on these advantages, Al5052 is one of the metals utilized in the proton exchange membrane fuel cell (PEMFC)[1]. PEMFC is an energy conversion system that turns chemical energy into electrical energy, where chemical reactions occur on both sides of the

anode and cathode membrane. The products produced by PMFC are electrical energy, as well as (313 – 353 K) hot steam and water, standards resistance coating for bipolar plate of DOE 2020 0.01 Ω/cm²[2], these circumstances affect the bipolar plate degradation produced by the acidic Nafion membrane. As a result, the risk of corrosion on the bipolar plate increases. The simulated acidic environment was 0.5 M of H₂SO₄ with a pH of 1–4[3,4,5]. Corrosion control is thus required to control corrosion damage.

Electrophoretic deposition (EPD) is metal coating

techniques deposits that uses deposits formed on the metal surface by moving particles in solution and moving to the metal surface due to an electric current that forms a thin layer protecting the direct contact of the metal with the environment, which is an option[6]. Furthermore, another issue in this study is the usage of inorganic chemicals as corrosion inhibitors such as chromate, arsenate, and others must be limited since they are hazardous and harm the environment.

Carbohydrate polymers are generally used as replacements for inorganic compounds in the control of the dissolution of the metals in aqueous media. Some of these are relatively high molecular mass compounds with polysaccharides properties[7]. In another previous study of Carbohydrate polymer for corrosion inhibitors, Arabic gum acts as a good inhibitor for the corrosion of API 5L X42 pipeline steel in 1 M HCl. Inhibition efficiency increases with the inhibitor concentration, and the maximum value is 92% at 2 g/L[8]. Arabic gum inhibitor was able to reduce the corrosion rate of aluminum metal immersed in an H₂SO₄ environment for 168 hours, the efficiency of the inhibitor was 79% at 0.5 g of Arabic gum inhibitor[9]. Research on mild steel, Inhibitor efficiency is 16.98% immersion time of 168 hours in an environment of 0.5 M sulfuric acid[10]. Another study showed that Arabic gum functioned as a mixed-type corrosion inhibitor on carbon steel in synthetic brine media, adsorption isotherm studies showed that the inhibitory film was mostly formed by chemisorption. Corrosion efficiency 90.5% with a concentration of 5 g. The results of Quantum chemistry calculations obtained highly branched polysaccharides with a molecular weight (MW) of 10330 g/mol. Polysaccharides rhamnose, arabinose, galactose, and glucuronic acid are adsorbed by chemisorption[11]. While the research was conducted on aluminum Al5052 with 0.5 g and 15-minute EPD coating in 0.5 M H₂SO₄ media, the efficiency obtained was 81%[12].

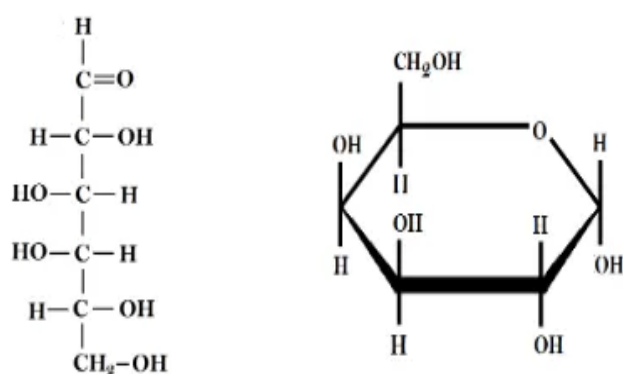


Fig. 1. Structure of d-galaktose

D-galactose is a simple sugar (monosaccharide) made from multi-fraction Arabic gum consisting of a highly branched polysaccharide dominand of 42% galactose, 27% arabinose, 15% rhamnose, 14.5% glucuronyl and 1.5% 4-O-methyl-glucuronosyl. besides that, d-galactose has the property of easily forming bonds with other monosaccharides to form Glucuronic acid-Galactose, Galactose-Arabinose, and pure Galactose which consists of a ring structure of 6 atoms formed due to the reaction of the (-OH) hydroxyl functional group on the C5 atom with the aldehyde on the C1 atom[10,11].

Aldehydes are good inhibitors of corrosion of mild steel in the three acids in which the aldehyde donates electrons and D-galactose its polymeric nature and tendency to dissolve in water[13]. In this work, aldehydes had been evaluated for their corrosion inhibition performance of D-galactose on Al5052 0.5 M sulfuric acid media with EPD coating technique with concentrate 0.5–1.5 g, with EPD time 15-45 minute. The corrosion inhibition performances were investigated using electrochemical techniques, *Fourier Transform Infrared (FTIR)*, EIS for resistance, and scanning electron microscopy (SEM).

2. Experimental and Procedures

2.1 Material Preparation

The sheet metal used in this study is aluminum alloy 5052 with a thickness of 2 mm and then is cut into a size of 20 x 10 mm. Then, the sheet metal is polished with sandpaper up to 2000 grit and pickling according to the ASTM G102 standard[14]. The coating process uses the electrophoretic deposition technique at a time variation of 15, 30, and 45 minutes with a voltage of 20 V.

2.2 Sample Preparation

The corrosion inhibitor used in this study was d-galactose with serial number 1.04062.0050 produced by Merck Germany with a concentration of D-galactose 0.5–1.5 g referring to previous research by Arwati et al. using Arabic gum in sulfuric acid media, it dissolved in distilled water up to 1000 ml[12]. The concentration of a corrosive solution was 0.5 molar H₂SO₄ pH 1–4.

2.3 Potentiodynamic Polarization Measurements

The corrosion rate test used a potentiodynamic polarization technique using a potential curve whose current was measured using a conventional three-electrode cell. The working electrode was Al5052, the reference electrode was AgCl[15], and the counter electrode was platinum. The potential range was -0.9 V to 1.5 V scan rate of 2 mV/s using a potentiostat

CS350. The calculation of corrosion rate and inhibitor efficiency are performed using equations 1 and 2 ASTM G102, respectively.

$$CR = K_1 \frac{I_{corr}}{\rho} \times EW \quad (1)$$

where:

CR : corrosion rate (mmPY),
K₁ : 3.27 x 10⁻³ (mm g/A.cm²),
I_{corr} : corrosion current (A/cm²),
P : density (2.68 g/cm³), and
EW : equivalent weight (9.05 g/mol).

$$IE = \frac{I_{blank} - I_{inh}}{I_{blank}} \times 100\% \quad (2)$$

where:

I_{blank} : current without inhibitor (A/cm²),
I_{inh} : current with inhibitor (A/cm²), and
IE : inhibitor efficiency (%).

2.4 Electrochemistry Impedance Measurements

The EIS test is displayed in the Nyquist plot in the form of a semicircular diagram which is a plot of the real impedance (Z') against the imaginary impedance (Z''). The resulting electrical parameters, called R_t, R_s, and CPE, were obtained based on the data fitting of the test results. The EIS tests were carried out at the OCP, with an amplitude of 10 mV and a frequency range from 0.01 Hz to 100 kHz. The EIS analysis was conducted at room temperature to be consistent with all the other tests.

$$IE = \frac{R_{inh} - R_{blank}}{R_{inh}} \times 100\% \quad (3)$$

where:

R_{blank} : resistance without inhibitor (kΩ/cm²),
R_{inh} : resistance with inhibitor (kΩ/cm²), and
IE : Inhibitor efficiency (%).

2.5 Fourier Transform Infrared (FTIR)

Fourier transform infrared spectrometer (Thermo-Fisher Scientific NICOLET IS10 FTIR spectrometer) with a spectral range of 500–4000 cm⁻¹. The samples on the aluminum substrate were prepared, then each sample was positioned on the holder sample. Measurement was conducted under room temperature.

2.6 Surface Morphology

The surface morphology test of Al5052 has been tested before and after D-galactose incorporation and cross section with a magnification of 500–3000 using

Scanning Electron Microscopy (SEM).

3. Results and Discussion

3.1 Result of Potentiodynamic Measurement

The effect of D-galactose on corrosion of Al5052 at different concentrations of 0.5–1.5 g EPD time 15–45 minutes in 0.5 M sulfuric acid at room temperature and the potentiodynamic polarization curve of Al5052 before and after being coated with a D-galactose inhibitor with a concentration of 0.5 and 1 g with variations times EPD 15, 30, and 45 minutes can be seen in Fig. 2 and Table 1. The curve shifts up when compared to Al without an inhibitor with a corrosion current value (I_{corr}) of 6.58 x 10⁻⁷ A/cm² and corrosion rate (CR) of 0.0075 mmPY.

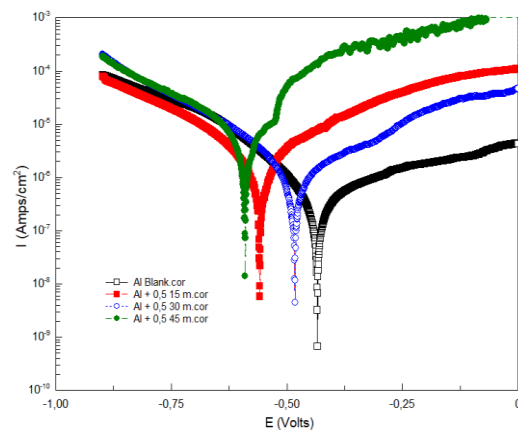


Fig. 2. Inhibitor concentration 0.5 g vs corrosion current

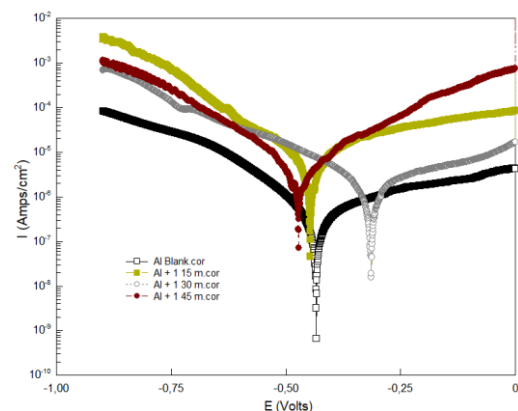


Fig. 3. Inhibitor concentration 1 g vs corrosion current

Fig. 3 depicts the highest corrosion current at 4.70 x 10⁻⁶ A/cm² CR value of 0.052 mmPY sample S4 and 5.25 x 10⁻⁶ A/cm² CR value of 0.0581 mmPY sample S7. This is because at a concentration of 0.5–1 g, the D-galactose layer on the surface of Al 5052 cannot be adsorbed. Due to the high solubility of d-galactose, precipitate does not form [6,12].

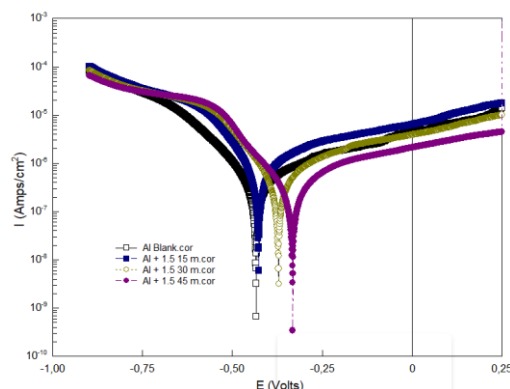


Fig. 4. Inhibitor concentration 1.5 g vs corrosion current

According to S8, which has a concentration of 1.5 g during an EPD time of 15 minutes, it can be seen in Table 1 and Fig. 4 that the I_{corr} and CR values are lower than S2–S7 instead of Al without an inhibitor which is $8.33 \times 10^{-7} \text{ A/cm}^2$ and 0.0091 mmPY. The short EPD time causes a layer of D-galactose on the surface of Al 5052 which is adsorbed inhomogeneously and dissolved by the environment [16], but different results are shown at a concentration of 1.5 g with an EPD time of 30–45 minutes, the polarization curve shifts downward, lowering the I_{corr} and the corrosion potential value (E_{corr}) in a positive direction indicated that the inhibitor is adsorbed in the anodic area and forms a layer on the metal surface that protects the metal from the environment. at a I_{corr} value of $3.75 \times 10^{-7} \text{ A/cm}^2$ on up to S10 [17].

Table 1. Potentiodynamic polarization results

Code	Sample/Minutes	E_{corr} (V)	β_a (mV)	β_c (mV)	I_{corr} (A/cm^2)	CR (mmPY)	EI (%)
S1	Al Blank	-0.434	576	171	6.85×10^{-7}	0.0075	-
S2	Al + 0.5g 15 m	-0.559	147	131	1.15×10^{-6}	0.0127	-
S3	Al + 0.5g 30 m	-0.482	453	196	2.01×10^{-6}	0.0222	-
S4	Al + 0.5g 45 m	-0.59	102	156	4.70×10^{-6}	0.052	-
S5	Al + 1g 15 m	-0.447	160	129	4.45×10^{-6}	0.0493	-
S6	Al + 1g 30 m	-0.314	103	300	4.83×10^{-6}	0.0534	-
S7	Al + 1g 45 m	-0.473	206	157	5.25×10^{-6}	0.0581	-
S8	Al + 1.5g 15 m	-0.427	297	122	8.33×10^{-7}	0.0091	-
S9	Al + 1.5g 30 m	-0.371	239	121	4.38×10^{-7}	0.0048	36
S10	Al + 1.5g 45 m	-0.332	290	154	3.75×10^{-7}	0.0041	45.2

Fig. 4 and Table 1 show that D-galactose is adsorbed through physisorption on the metal surface as the inhibitor's effectiveness improves with increasing concentration and time of EPD, in a concentration of 1.5 g, the inhibitor efficiency was 36% for 30 minutes and 45.2% for 45 minutes, respectively. When compared to research by Arwati et al., 2020, with the same parameters 0.5–1.5 g EPD for 15–45 minutes, Arabic gum is still better than pure

D-galactose for inhibiting the metal surface of al 5052 in 0.5 M sulfuric acid medium with an inhibitor efficiency until 81% [12].

3.2 Result of Fourier Transform Infrared (FTIR)

FTIR spectra D-galactose 1.5 g on aluminum alloy substrate in three variation of deposition times, 15 min (sample C), 30 min (sample B) and 45 min (sample A) is shown in Fig. 5.

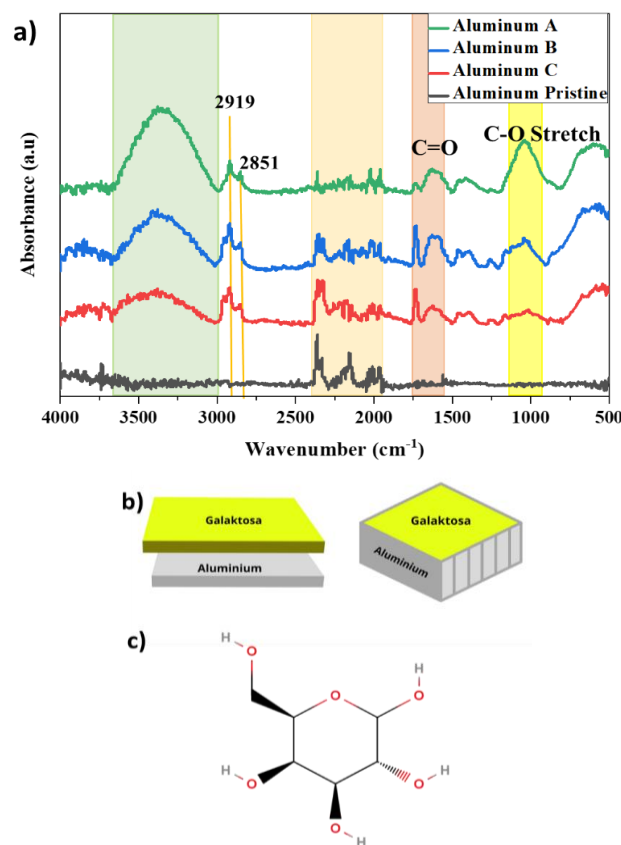


Fig. 5. FTIR spectra and deposit layer of Al5052

The characteristic peaks of D-galactose do not appear on the pristine aluminum substrate FTIR spectrum. Upon deposition of D-galactose for 15 minutes, the broad peak appeared in the range of $3000\text{--}3600 \text{ cm}^{-1}$, which refers to the formation of hydrogen bonding of hydroxyl group [18]. This can be assigned to the chemical structure of D-galactose. Methyl group of D-galactose also appear on 2918 cm^{-1} and 2850 cm^{-1} which attributed to $=\text{CH}_2$ asymmetric stretch and $-\text{CH}_3$ symmetric stretch, respectively [19]. Carbonyl group on $1500\text{--}1700 \text{ cm}^{-1}$ represent $\text{C}=\text{O}$ bond of amide, and aldehyde [20]. Peak $1097\text{--}1035 \text{ cm}^{-1}$ which attributed to $\text{C}-\text{O}$ were connected to the secondary and primary alcohols, respectively [21].

Based on these IR-sensitive features, the D-galactose was successfully deposited on the substrate. As we incrementally changed the deposition time, the

intensity of the corresponding characteristic peaks of D-galactose are increasing. For example, the hydroxyl group and C-O stretch peaks exhibit an increasing of intensity in accordance with the addition of deposition time. It can be concluded that the longer the deposition time of D-galactose on the aluminum substrate resulted the intensity enhancement of D-galactose characteristic IR peaks[22], the sugars with hydroxyl and aldehyde groups have the least ability to donate electrons, so a large enough concentration is needed to precipitate it on the surface of Al 5052[11].

3.3 Result of EIS Measurement

A semicircular Nyquist plot along the real axis is shown in Fig. 6 and Table 2, which demonstrates that the ion diffusion mechanism happens at the electrode during the test when a D-galactose inhibitor is added. The semicircular diameter of Al5052 after EPD is higher than that of Al5052 before EPD, suggesting the adsorption of inhibitor molecules on the metal surface as a result, the corrosion current is reduced[11]. The resistance value for Al after 30 minutes of EPD with a concentration of 1.5 g the value of R_s 4.92 Ω/cm^2 , R_t 1.8 $k\Omega/cm^2$, for Al after 45 minutes of EPD with a concentration of 1.5 g a resistance value increased to 5.73 Ω/cm^2 R_t 2.2 $k\Omega/cm^2$ while Al5052 before EPD resistance value R_s 4.86 Ω/cm^2 , R_t 1.2 $k\Omega/cm^2$.

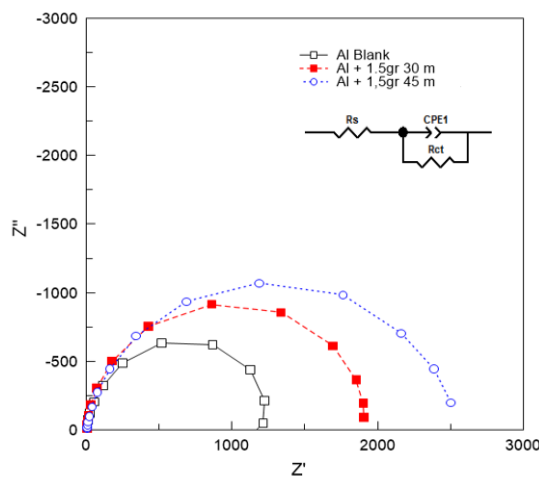


Fig. 6.. Nyquist Plot of Al5052 before and after EPD

Table 2. Impedance results test

Samples/Minutes	R_s (Ω/cm^2)	Cpe	R_t ($k\Omega/cm^2$)	EI (%)
Al Blank	4.86	1.96×10^{-6}	1.2	-
Al + 1.5g 30 m	4.92	2.35×10^{-6}	1.8	33.3
Al + 1.5g 45 m	5.73	2.54×10^{-6}	2.2	45

In the presence of D-galactose inhibitors, the inhibitor raises the value of resistance or charge

transfer (R_t) and forms a layer that impedes the transfer of charge or electrons from the metal surface to the solution medium, resulting in a decrease in the oxidation and reduction reactions of ions in solution[12]. The highest resistance efficiency value is only 45%. With an increase in resistance of 1000 Ω/cm^2 , it does not meet DOE 2020 and 2025 standards for bipolar plate PEMFC. Because the coating resistance value is more than 0.01 Ω/cm^2 [2,23].

3.4 Result of Cross-section Scanning Electron Microscopy

Fig. 7.(a) depicts the SEM morphology of the blank coupon before the electrochemical experiment. The surface of Al5052 looks like a straight line, and the light shows that there is no adsorbed layer on the surface of Al5052. The darker color of the grooves implies that there is a layer covering the surface of Al5052, the hydroxyl groups and C=O aldehyde bonds[22]. These results clearly show that the hydroxyl and aldehyde groups had the electron donating ability by physically on the surface of Al5052[11] as shown in Fig. 7(b).

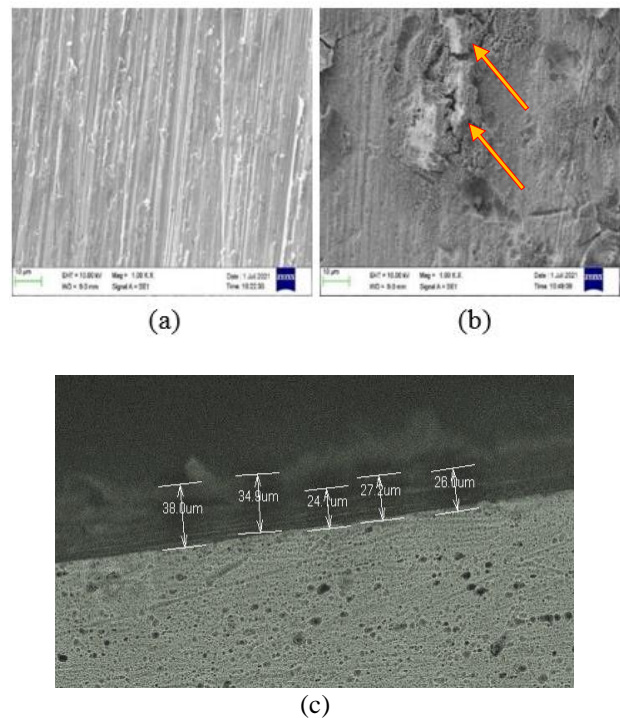


Fig. 7. Surface morphology Al5052 (a) Al5052 blank, (b) Al5052 with 1.5 g D-galactose, and (c) cross-section Al5052 1.5g D-galactose

Fig. 7(c) a shows EPD can deposit particles on the surface of materials with thicknesses ranging from 1.0 to 100 μm [6], a thickness of the Al5052 layer is an average of 29.8 μm . This value is very thick if



comparison research by Arwati et al., 2020. Arabic gum layer for Al5052 reached 12.15 μm . The thickness of the material coated is very important because thickness of the layer affects performance of the bipolar plate[24].

3.5 Result of Surface Morphology

Characteristics of morphologies between before and after EPD with 1.5 g inhibitor for 45 minutes through electrochemical methods can be found in Fig. 9 and Fig. 10. Throughout before EPD's SEM data, the sample exhibited general corrosion with the onset of pitting[25].

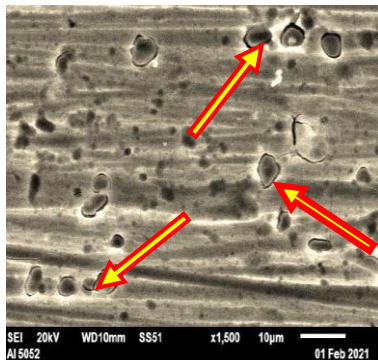


Fig. 9. Surface morphology Al5052 blank

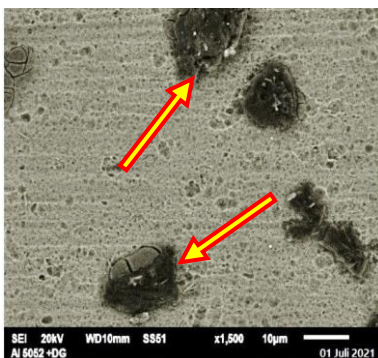


Fig. 10. Surface morphology Al5052 with 1.5 g inhibitor

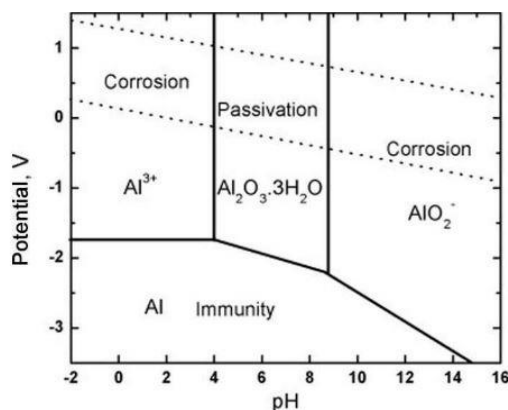


Fig. 11. Pourbaix diagram for aluminum

This difference can be explained based on thermodynamic considerations. Fig. 11, Al-containing alloys are nominally passive at pH 7 as a result of a protective Al_2O_3 film formation. However, pH 1-4 PEMFC under acidic and basic conditions, this protective film becomes soluble, and its continuity is disrupted, resulting in the dissolution of the alloy surface. In acidic conditions, Al is oxidized to Al^{3+} [26,27]. While SEM images of Al5052 after EPD with 1.5 g and period of 45 minutes revealed a smooth surface and visible black lumps indicating that the D-galactose inhibitor adsorbed on the metal surface shield Al5052 from the corrosive environment [12,28]. The outcomes of SEM morphology and electrochemical method are similar as well as determined inhibitor efficiency is 45.2%.

4. Conclusions

The conclusions in this study are the results of electrochemical analysis, the best inhibitor concentration is at 1.5 g and the EPD time is 45 minutes with a resistance value increase with increasing EPD time and inhibitor concentration where the Al value after 30 minutes EPD concentration is 1.5 g. After 45 minutes of EPD and a concentration of 1.5 g, the resistance value for Al increased to $5.73 \Omega/\text{cm}^2$ and R_t $2.2 \text{ k}\Omega/\text{cm}^2$, whereas the resistance value for Al5052 before EPD was R_s $4.86 \Omega/\text{cm}^2$ and R_t $1.2 \text{ k}\Omega/\text{cm}^2$. The corrosion rate of Al5052 before EPD with D-galactose inhibitor was 0.0075 mmPY while the corrosion rate of Al5052 after EPD was 0.0041 mmPY with an inhibitor efficiency value of 45.2%. Upon deposition of D-galactose for 15 minutes, the broad peak appeared in the range of $3000\text{--}3600 \text{ cm}^{-1}$, which refers to the formation of hydrogen bonding of hydroxyl group. This can be assigned to the chemical structure of D-galactose. Methyl group of D-galactose also appear on 2918 cm^{-1} and 2850 cm^{-1} which attributed to $=\text{CH}_2$ asymmetric stretch and $-\text{CH}_3$ symmetric stretch, respectively. Carbonyl group on $1500\text{--}1700 \text{ cm}^{-1}$ represent $\text{C}=\text{O}$ bond of amide, and aldehyde. Peak $1097\text{--}1035 \text{ cm}^{-1}$ which attributed to $\text{C}-\text{O}$ were connected to the secondary and primary alcohols. While the results of surface morphology with SEM Al5052 before EPD showed pitting corrosion. In contrast, SEM Al5052 after EPD exhibited a smooth surface and visible black lumps indicating that the D-galactose shield Al5052 from a corrosive environment.

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