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Effect of Organics Corrosion Inhibitors on the Corrosion of 304SS in 3.5% NaCl

F. Gapsari, R. Soenoko, A. Suprapto, W. Suprapto

Abstract – This work is a preliminary study with the purpose to find some information about the effectiveness of Ceraflava (CF) extract as inhibitor in 3.5 %NaCl, CF or yellow bees wax from honey farm used as corrosion inhibitor. In particular the purpose of this study is to determine the ability of CF in inhibiting the corrosion rate. The inhibition of the CF extract on304SS in 3.5% NaCl is carried out with potentiodynamic polarization, EIS and SEM. The findings show that CF can be used as corrosion inhibitor in SS 304 even though CF can only be categorized as moderate performance of inhibitors. However, it was observed that inhibitors can be adsorbed on the 304SS surface so it inhibits the corrosion rate. Based on the potentiodynamic polarization and EIS results, the inhibition efficiencies are 59.77% and 53.84% in 0.2g/L inhibitor concentration. Potentiodynamic polarization curves indicate the mixed type of the inhibitors.

The adsorption of the inhibitors on the 304SS surface was found to obey to Langmuir adsorption isotherm. Thermodynamic adsorption parameter were calculated and discussed. It is expected this research may be recommended in the use of CFCs in saline solution or other solutions on the science of corrosion controlling using organic inhibitors. Copyright © 2016 Praise Worthy Prize S.r.l. - All rights reserved.

Keywords: 304SS, Electrochemical Technique, Inhibitors, NaCl Solution, SEM

I. Introduction

Type 304SS is the most versatile and widely used of all the stainless steels [1]-[43]. The 304SS has a good corrosion resistance [1]. The 304SS is the type of stainless steel mainly used for canned food, kitchen utensils, medical devices and automotive industry [2]. However, the disadvantage of this type is that it stimulates local corrosion when exposed to chloride or located in the environment containing chloride (Cl⁻) ion [3]. The environments with the most frequent contacts with metal are water, environment containing the Cl-ion and acid environment. Chloride ion is the most aggressive ion to cause metal corrosion especially on the 304SS. However, the resistance to pitting corrosion of 304SS in solutions containing Cl⁻ is not good enough and it adversely influences both the service life and integrity of structures made by this material [1]. Some studies on the 304SS corrosion have been widely conducted [1], [4], [5]. Similarly, many efforts have been taken in order to increase the 304SS resistance towards NaCl [6]-[9]. The inhibitor is the most effective method in preventing the corrosion of metal surfaces by aggressive solutions. Organic compounds act as a good inhibitors due to their heteroatom structures such as sulfur, nitrogen and oxygen [10]–[14]. Various organic compounds have been studied as corrosion inhibitors for 304SS in different solution [15]-[17].

In the present study, Ceraflava (CF) or yellow bees wax by honey farm is used as corrosion inhibitor.

CF is generally used as a remedy for dry lips or skins. It contains flavanoid and antioxidant. The aim of this paper is to study the effectiveness of the inhibitor addition on the 304SS in 3.5% NaCl solution.

II. Materials and Methods

II.1. Cera Flava Extraction

After the honey and the honeycomb were separated, the remaining is CeraFlava.200 grams of CF were mashed and extracted with 200 ml of 99% ethanol (Merck). The mixture was filtered and then refluxed for 4 hours at 50° C.

II.2. FTIR Testing

Shimadzu IR Prestige-21 type of Fourier transform infrared (FTIR) spectrometer instrument was used with the following resulting characteristics: IR-spectral in range 4000-400 (cm⁻¹) with 4 cm⁻¹ resolution and KBr matrix with a scan rate of 32 scans per minute.

The functional groups in CF extract are determined by FTIR.

II.3. Materials

The chemical composition (wt. %) of 304SS is 0.04%C, 0.92 % Mn, 0.52 % Si, 0.030% P, 0.002 % S, 18.15 % Cr, 9.58% Ni and Bal.Fe.

The density of 304SS was 7.9 g/cm³. The 304SS was added to epoxy resin with a geometric exposed surface area measuring 1 cm^2 and connected to the electrolyte.

Before the experiments, the specimens were polished with 200, 400, 600, 1000, 1500, and 2000 grades of SiC paper.

II.4. Electrochemical Measurement

Corrosion testing was carried out using the G-31 American Standard and Testing (ASTM) [18].

The variable used in the study is an independent one in the form of inhibitor concentration, namely 0, 0.1, 0.2, 0.3, and 0.4 g/L of CF extract.

The room temperature was 25° C. The electrochemical testing was carried out with the PGSTAT 128N AUTOLAB device. The prepared specimen was made into an electrochemical cell with platinum as the counter electrode (CE), Ag/AgCl (KCl 3 M) as the reference electrode (RE), and304SSspecimen as the working electrode (WE). The three electrodes were immersed in batch for 3 hours. The measurement of polarization in -2 V until 1.5 V range corrosion potential (OCP) with scan rate has given a value of 20 mV/s. The inhibition efficiency was measured with the following Eq. (1) [19]:

$$EI(\%) = \frac{Icor - Icor(i)}{Icor} \times 100\%$$
(1)

where I_{cor} and $I_{cor(i)}$ indicate the corrosion current density with and without addition of inhibitors.

The *Electrochemical Impedance Spectroscopy* (EIS) measurement was carried out at the frequency of 100 kHz to 10 MHz and 5 mV as amplitude using AC signal at Open Circuit Potential (OCP). The inhibition efficiency with the EIS method was formulated with the following Eq. (2) [29]:

$$EI(\%) = \frac{R_{ct(i)} - R_{ct}}{R_{ct(i)}} \times 100$$
 (2)

III. Result and Discussion

III.1. FTIR Analysis

Based on the FTIR testing, the function of the CF extract can be found. Fig. 1 shows the function of the CF extract (see Fig. 1). The result of FTIR analysis is shown in Fig. 1. There are some absorbed spectrum with high intensity at some wave numbers, namely 3369.41 cm⁻¹ that is detected as the absorption of phenol functional group. The absorption of CH alkane appears at 2925.81 cm⁻¹. The CC alkene is shown at 1618.17 cm⁻¹.

The other absorption are shown at 1454.23 cm⁻¹ of the CH alkane functional group, and 1049.20 cm⁻¹ as the CO alcohol functional group. It shows that the CF extract has C - H, C = O, C = C, and O - H as its functional groups.

The functional groups match phenol and aromatic compounds.



Fig. 1. Result of FTIR Testing of CF Extract

III.2. Potentiodynamic Polarization Studies

Polarization testing was carried out towards CF extract concentration variation. The results of inhibition efficiency measurement using the polarization method are presented in Table I.

Polarization testing was carried out towards CF extract concentration variation. The results of inhibition efficiency measurement using the polarization method are presented in Table I. Fig. 2 shows tafel plots with various concentration. The optimum inhibition efficiency obtained was 59.77% by addition of 0.2 g/L inhibitor.

TABLE I Inhibition Efficiency Measurement With The Polarization Method

| CF extract | β_a | B_c | E_{corr} | Icorr | IE | | |
|------------|-----------|---------|------------|-----------------------|-------|--|--|
| (g/L) | (V/dec) | (V/dec) | (V) | (A/cm^2) | (%) | | |
| 0 | 0.156 | 5.440 | -0.341 | 2.61×10-6 | 0 | | |
| 0.1 | 0.109 | 0.149 | -0.378 | 1.19×10 ⁻⁶ | 54.40 | | |
| 0.2 | 0.140 | 0.194 | -0.370 | 1.05×10-6 | 59.77 | | |
| 0.3 | 0.085 | 0.180 | -0.198 | 1.53×10-6 | 41.37 | | |
| 04 | 0.124 | 0.302 | -0 344 | 1.62×10^{-6} | 37.93 | | |



Fig. 2. Tafel plots with various concentration of CF extract in 3.5% NaCl

The inhibition efficiency increased at 0.1 to 0.2 g/L addition of inhibitors. Then, the efficiency decreased at 0.3 g/L addition of inhibitors. The addition of inhibitors as much as 0.1; 0.2 g/L, the corrosion potential (Ecorr) show a tendency to anodic (Fig. 2). It shows that the CF extract controls the anodic reaction by forming complex molecules in the anodic area on the 304SS surface [20].

On the other hand, change into cathodic occurred with the addition of 0.3 g/L. The cause of the change was the formation of complex molecules in the cathodic area of the 304SS surface [20]. The Ecorr values of the solution with inhibitors increased and decreased randomly compared to a solution without inhibitors. It indicates that CF extract is categorized as mixed type inhibitors [21], [22]. The optimum inhibition efficiency occurred in 0.2 g/Land decreased with the addition of inhibitor concentration; a possible cause of the tendency can be physisorption. The more addition of CF extract caused a saturated solution and released the protective film so that the efficiency decreased. CF extract which was added and functioned as the inhibitors and had optimum concentration, when added beyond its optimum concentration, will saturate and may cause formed layer separated. This was caused by the interaction among inhibitor molecules larger than inhibitor interaction on the metal surface. The inhibition efficiency decreased at higher concentration caused by desorption molecules from the metal surfaces [23], [24]. The saturated solution initiated interaction between metal and surface stopped and caused the metal surface not to be covered therefore decreasing the inhibition efficiency.

The change of anodic (βa) and cathodic (βc) tafel constants is irregular (Table I). The irregularity of βc shows that the inhibitor is not optimum to decrease the cathodic reaction in the metal [25].On the other hand, the irregularity of βa value indicates that there is physisorption the metal surface.

The CF extract has a moderate inhibition capacity in all the variations of concentration. The inhibitor capacity to decrease the corrosion is different from another depending upon its ability to form complex molecules. Upon the relative solubility, the result can either inhibit or catalyze the further dissolution of metal [26]. The inhibition is definite and depends on the characteristics of metal and corrosive environment [27]. Meanwhile, the corrosion inhibitors are opted for inhibited solubility of the fluid [28]. The immediate absorption from the functional group forces the molecules of CF extract to move horizontally using the surface of orientated metal.

The immediate absorption causes each functional group interact on its own.

The corrosion rate of 304SS NaCl solution is set off through two main procedures, namely formation and build-up of an iron oxide layer by pitting destruction [29]. Due to the corrosion mechanism of stainless steel in a saline mode, at the anode regions, stainless steel has the following potential dissolution reaction: Fe \rightarrow Fe²⁺ + 2e⁻, occurring adjacently to the surface of the cathode reaction: $1/2O_2 + H_2O + 2e^- \rightarrow 2OH^-$ [8]. The missing inhibitor brings about an active behavior, without the formation of a passive layer showing offensive formation of corrosion product layer. When inhibitor is added, there is a tendency that the Ecorr values move toward more active value while the Icorr values decrease. The anodic shows an active behavior, similar to what is observed for non-inhibitor solution. The behavior might be caused by the absence of inhibitor that dissolves in water [30]. The inhibitor may not be able to be put directly into the corrosive solution.

In cathodic, the current density is limited due to the oxygen reduction. It can be seen that the addition of inhibitor has no significant effect in the cathodic at polarization curve because the limitation of current density. The change to the cathodic occurs only at the addition of 0.3 g/L as the inhibitor after surpassing the optimum concentration to increase inhibition efficiency.

There is a possibility that it causes the maximum inhibition capacity at 59.77%.

III.3. Electrochemical Impedance Spectroscopy (EIS)

The impedance diagrams represented in Nyquist plot obtained at OCP are presented in Fig. 3. The parameters of the EIS testing are described in Table II. They are the transfer resistance of R_{ct} , the solution resistance (R_s) and the Constant Phase Element(CPE).The interaction between the surface of metal and the solution causes a transfer charge between them measured as R_{ct} .

The corrosion behavior of 304SS frequency in different variations of solution concentration is shown by the Nyquist plot as seen in Fig. 3. The partial circle shows the characteristics of solid electrode and thas irregular and non-homogonous electrode [31], [32].

The Nyquist plots of 304SS at the various concentration of inhibitor exhibit similar patterns. This occurrence specifies the inhibition mechanism (a charge-transfer process between the inhibitor and the metal surface) and it is also alike [15]. In this case, the result of the EIS test shows similar result to the potentiodynamic polarization with the highest inhibition efficiency at the concentration of 0.2 g/L (Section III.1).

TABLE II Impedance Parameter Of The 304SS In 3.5% NACI

| In EDA CE TAICAMETER OF THE 50 155 IN 55 /0 TOTEE | | | | | | | |
|---|-----------------|----------------|---------|----------------|-------|--|--|
| CF extract | R _{ct} | R _s | CPE | ~ ² | IE | | |
| (g/L) | (Ω) | (Ω) | (µF) | χ | (%) | | |
| 0.0 | 39.924 | 1.012 | 154.410 | 0.036907 | 0 | | |
| 0.1 | 72.868 | 1.212 | 68.437 | 0.011062 | 45.21 | | |
| 0.2 | 86.487 | 1.877 | 57.212 | 0.037338 | 53.84 | | |
| 0.3 | 62.178 | 1.779 | 68.166 | 0.009212 | 35.79 | | |
| 0.4 | 52.691 | 1.471 | 65.258 | 0.015423 | 24.23 | | |



Fig. 3. The Nyquist plot of the 304SS in 3.5%NaCl with the cf extract concentration variation

Fig. 4 shows the corresponding circuit diagram used to fit the EIS in which the calculation of the impedance parameters was simulated using the corresponding circuit. The interaction between the metal and the solution was illustrated.



Fig. 4. Equivalent circuit used to fit the EIS diagrams

III.4. Adsorption Isotherm

The mechanism of the corrosion protection may be explained on the basis of adsorption behavior [33].

The interaction information between the inhibitor molecule and metal surface can be provided by the adsorption isotherm [34]. Generally, organic molecules can prevent the corrosion on metal using the adsorption. The type of adsorption depends on chemical molecule composition, temperature and electrochemical potential of the metal or interface between solution and metal. The measurement of isothermal adsorption of the findings is adjusted to match various isothermal equations, namely Frumkin, Langmuir, Temkin, Freundlich, Bockris-Swinkelsand Flory-Huggins [35], [36]. The best fitted result of the study follows the Langmuir isothermal adsorption (equation (3)) shown in Fig. 5:

Langmuir's Equation:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \tag{3}$$

where $\theta = \frac{EI}{100}$, $\theta =$ surface coverage and $K_{ads} =$ adsorption equilibrium constants.



Fig. 5. Langmuir adsorption isotherm

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The free energy of adsorption (ΔG_{ads}°) was calculated by the equilibrium constant of adsorption (K_{ads}) using Eq. (4):

$$\Delta G_{ads}^{\circ} = -2.303 RT \log(K_{ads} \times 1000) \tag{4}$$

where 1000 is the concentration of water in the solution (g/L), R is the universal gas constant and T is the absolute temperature.

The values of free energy are negative as well as less than - 40 kJmol⁻¹, indicating the spontaneous physical adsorption of the inhibitors on the metal surface [37].

It gives the same conclusion as the findings of polarization and EIS. The negative values of G_{ads} showed that the adsorption of inhibitor molecules on the surface occur spontaneously [38]. The calculation of the enthalpy value (ΔH_{ads}°) is the Van't Hoff's formula in Eq. (5):

$$\ln K_{ads} = \frac{-\Delta H_{ads}^{\circ}}{RT}$$
(5)

So that the entropy adsorption (ΔS_{ads}) can be measured using Eq. (6):

$$\Delta G_{ads} = \Delta H_{ads}^{\circ} - T \Delta S_{ads}^{\circ} \tag{6}$$

Therefore, the thermodynamic parameter of inhibitor adsorption can be seen in Table III.

The positive value of ΔH_{ads}° shows that the process of dissolution is exothermic, while, the positive value of ΔS_{ads}° shows that the adsorption process followed with an increase in the entropy, which is the driving force for the adsorption inhibitor to the metal surface [39].

III.5. SEM Analysis

The results of SEM are illustrated in Figs. 6 showing micro photograph of the specimen without inhibitor and specimen with the CF extract inhibitor with 0.2 g/L concentration. Figs. 6(a) and 6(b) show the images of the specimen without and with inhibitor, respectively.

On Fig. 6(a), it can be seen a small hole on the 304SS surface. Meanwhile, the solution with inhibitor is smoother even though there are still small holes that indicate corrosion.

Therefore, there should be a synergic effect effort to improve the performance of inhibitor.



Figs. 6. Scanning Electron Microscopic images of 304SS (a) absence inhibitor (b)presence inhibitor in 3.5% NaCl

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IV. Conclusion

Based on the results, the CF extract as corrosion inhibitors works by physisorption. The inhibition capacity of the CF extract is relatively low.

The highest inhibition efficiency occurs in the addition of 0.2 gr/L of CF extract as much as 59.77% and 53.84% with the potentiodynamic polarization and EIS measurement, respectively.

The electrochemical testing showed that the CF extract can carry out adsorption on the 304SS surface, but it is unable to form a protective layer either simultaneously or thoroughly on the surface. There should be more efforts for synergistic effects so that the performances of inhibitor will increase.

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