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THE INHIBITION OF 304SS IN HYDROCHLORIC ACID SOLUTION BY *CERA ALBA* EXTRACT

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Abstract

Organic inhibitors became popular for protection against corrosion especially in acid solution in acid to prevent metal dissolution. Hydrochloric acid is widely used in the pickling, cleaning, and descaling process of metals. The high performance of corrosion inhibition efficiency of *Cera alba*(CA*)* extract was investigated for 304SS corrosion in 1Mhydrochloric acid using polarizationpotentodynamic method. The extract of CA was characterized by Fourier Transform Infrared Spectroscopy (FTIR). The polarization study indicated that the formulations act as mixed-type inhibitor. The adsorption process on 304SS followed Frumkin isotherm. The associated activation parameters and the thermodynamics data of adsorption were evaluated and discussed. The adsorbed film formed on the metal surfaced was characterized byScanning Electron Microscope (SEM).

Keywords: Cera alba, EIS, FTIR, Inhibitor, Polarization.

1.Introduction

Stainless steel type 304 is the most common form of stainless steel used in many industrial applications involving in tank production and other appliances connected to chemical material, petrochemical, paper, pollution control, hydrometallurgy and petroleum industry. In several producing activities at high temperature, some companies begin to use 304SS. It is reccomended to use in furnaces, refinery petrochemical, super heater, hanger tubes, radiant tubes, anchors refractory, dampers, inner tubes, lifters, expansion bellow, furnace fan, muffle, and bell furnaces. Corrosion of metals in aggressive solutions is an important problem in industrial cleaning and pretreatment processes such as acid

Nomenclatures

A	Arrhenius
C	Concentration of inhibitor, g/L
CR	Corrosion rate, mmpy
Ea	Activation energy, kJ/mol
E_{corr}	Corrosion potential, V
h	Planck's constant
I_{cor}	Corrosion current density of absence inhbitor, A/cm^2
$I_{cor(i)}$	Corrosion current density of presence inhbitor, A/cm^2
IE	Inhibition efficiency, %
K_{ads}	Adsorption equilibrium constant
N	Avogadro's number
R	The universal gas constant
R^2	Coefficient of determination
T	Temperature, K

Greek Symbols

α	interaction parameter
βa	Cathodic tafel slopes, V/dec
βc	Anodic tafel slopes, V/dec
ΔS	Entropy, J/mol.K
ΔG_{ads}	Free energy of adsorption, kJ/mol
ΔH	Entalpy, kJ/mol
θ	Degree of surface coverage

Abbreviations

AR	Acid Reagent
ASTM	The American Society for Testing and Materials
ads	Adsorption
CA	Cera Alba
CE	Counter Electrode
FEI	Field Electron and Ion
FTIR	Fourier Transform Infrared
HCl	Hydrochloric Acid
OCP	Open Circuit Potential
RE	Reference Electrode
SEM	Scanning Electron Microscope
SS	Stainless Steel
WE	Working Electrode

pickling, industrial acid cleaning, acid descaling and oil well acidizing. Corrosion inhibitor is a popular method to prevent and protect corrosion rate of metal surfaces which are in an acidic environment. [1, 2]. By adding small concentration of these materials to corrosive environments, will prevent the reaction of metal with media[3-7]. Inhibitors reduce the corrosion rate by changing the anodic or cathodic reaction, reducing diffusion rate of reactants into the metal surface [8]. The inhibitors are derived from inorganic and organic compound in which aninorganic compound isexpensive and not environmentally friendly [9, 10]. Therefore, the study of new non toxic inhibitors is important to solve this problem. The study of organic corrosion inhibition is an alternative field of research due to its usefulness in various industries. These compounds act as good inhibitors due to their heteroatom structures such as sulfur, nitrogen and oxygen [11, 12].

Some research on organic inhibitor in hydrochloric acid have been studied [2, 13-19].Nonetheless, there is still a need for research on other organic compounds can be used as inhibitors in industrial applications.

This present work therefore introduces Cera Alba (CA) extract as a novel green inhibitor for 304 SS in hydrocloric acid. CA is a natural wax produced by honey bees. CA is used to store honey, royal jelly, and propolis. Honey is a natural material that has been reported good for retarding the corrosion rate of various metal [20-23]. The same with propolis [24, 25]. The objective of this study was to investigate the effect of CA extract as corrosion inhibitors for 304SS in M HCl. Potentiodynamic polarization method were utilized, and adsorption isotherms and thermodynamic parameters were calculated and discussed.

2. Experimental Method

Experimental method is begun by preparing the metal specimen and the inhibitor. Later, the extraction from the next inhibitor is characterized. The aim is to find out the content of the inhibitor. Next, the inhibitor is added to the solution which has been prepared before the electrochemical test.

2.1. Preparation of specimens

The experimet were perfomed with 304SS with the following composition (wt %): 0.04%C, 0.52 % Si, 0.92 % Mn, 0.030% P, 0.002 % S, 9.58% Ni, 18.15 % Cr, Bal.Fe. Density of 304SS was 7.9 g/cm^3 The 304SS was added to epoxy resin with a geometric exposed surface area measuring 1 cm^2 and connected to the electrolyte. The 304SS were polished consecutively with emery paper of from 500 to 1500 grades. The specimen were washed with distilled water, rinsed in ethanol, degrased with acetobe and finally dried.

2.2. Inhibitor preparation

CA used was obtained from Malang, Indonesia. The CA was extracted by solid-liquid method in order to obtain the optimum conditions. Half of CA powder was extracted by maceration method using 99% ethanol (Merck). 200 g of CA was extracted with 200 ml of ethanol. The ethanol was evaporated by using a water bath.

2.3. Inhibitor characterization

FTIR analysis of CA extract was used to determine the functional groups. Infrared spectra were recorded in Shimadzu IR Prestige-21, FTIR spectrometer. This spectrometer using KBr-disc techniques. The region between 4000-400 cm^{-1} with resolution of 2 cm^{-1}.

2.4. Solution preparation

The corrrosive media of 1 M HCl was prepared by dilution of AR grade 37% HCl (Merck) using distilled water. HCl was used as corrosive media to explore potential inhibition of CA extract.

2.5. Electrochemical measurement

Corrosion testing conducted based on standards ASTM (the American Society for Testing and Materials) G-31 [18] (ASTM). To know kinetic of corrosion process polarization potentiodynamic were used. Independent variables used in this study were 0, 1, 2, 3, and 4 g / L CA extract concentration. In this case, the room temperature was set at 25^0C and the electrochemical measurements were performed with the Autolab PGSTAT 128N. A-three-electrode system was used to perform electrochemical tests which include 304SS as working electrode (WE), Ag/AgCl (KCl 3 M) as reference electrode (RE), and platinum as counter electrode (CE). Three electrodes immersed in a batch for 2 hours.

The starting potential was -0.1 V and the scan rate range extended up +0.1 V at Open Circuit Potential (OCP) with voltage scan rate of 0.001 V/s. Inhibition efficiency calculated by Eq. 1 [26, 27]:

$$IE\ (\%) = \frac{Icorr - Icorr(i)}{Icorr} \times 100\ \% \qquad (1)$$

where, $I_{corr}$and $I_{corr(i)}$ is corrosion current density in case of absence and presence inhbitor.

2.6. Scanning electron microscope (SEM)

SEM type FEI, inspect type -S50 was employed to analyzed surface morphology of 304SS at various concentration of inhibitor. The 304SS specimens was prepared as described at section 2.1.

3. Result and Discussion

The result of the experiment test will be analyzed in detail in this section. The result of CA extract characteristic can be seen from the result of FTIR. The corrosion rate and efficiency inhibition are diplayed on Fig. 1 and Table 1.

3.1. Fourier transform infrared spectroscopy (FTIR)

FTIR testing results showed some absorption spectrum with strong intensity. The strong absorption band at 3369.41cm^{-1}is atributed to O-H.Wave number2925.81 cm^{-1}is related to C-H. a carbonyl group (C=O, ketone) at 1714.6 cm^{-}1. Aromatic group at 1618.17 cm^{-1} and 1454.23 cm^{-1}. The absorption band at 1454.23 cm^{-1} is related to C-H. As well as the wavenumber 1049.20 cm^{-1} as the absorption of the alcohol functional group C-O. The result implies that CA extract have flavanoids content.

3.2. Potentiodynamic polarization method

Potentiodynamic polarization method aims to determine the effect of extracts CA inhibition and types of inhibitor. Tafel plot of 304SS in HCl media with various concentrations of inhibitors shown in Fig. 1. The IE (%) was calculated using the Eq. (1). The electrochemical parameters obtained from the polarization measurements are listed in Table 1.

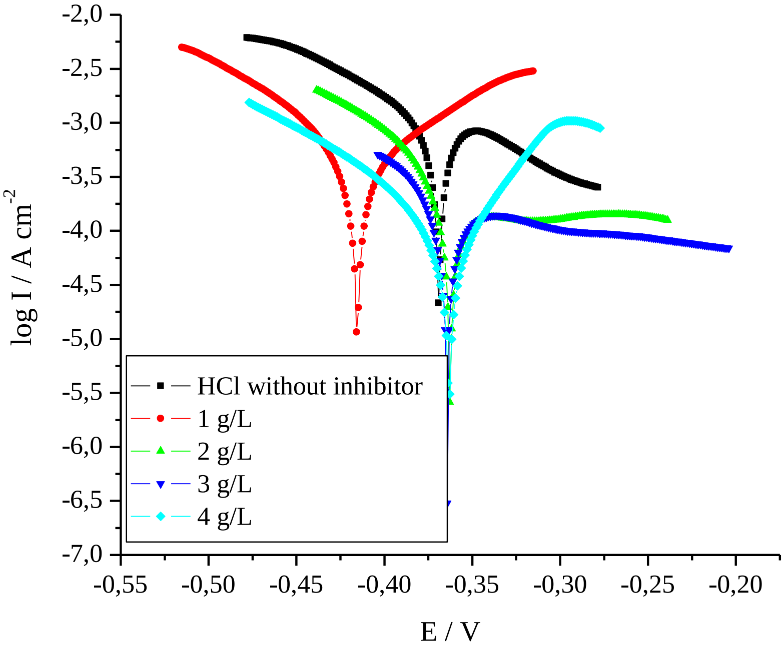

Fig. 1. Tafel plots with various concentration of CA extract.

Table1. Tafel extrapolation of 304SS in 1M HCl in various concetration of CA extract.

Inhibitor (g/L)	*βa* (V/dec)	*βc* (V/dec)	E_{corr} (V)	I_{corr} (A/cm^2)	*CR* (mmpy)	*IE* (%)
0	0.086	-0.5171	-0.37	2.90×10^{-3}	30.17	-
1	0.179	-7.844	-0.42	2.33×10^{-3}	24.26	19.6
2	0.052	-0.10	-0.36	6.55×10^{-4}	6.88	77.42
3	0.071	-0.116	-0.33	2.80×10^{-4}	2.91	90.34
4	0.072	0.658	-0.36	9.63×10^{-7}	1.001	99.96

Polarization measurement results as shown in Table 1 indicate the effect of CA extract to corrosion inhibition on 304SS in hydrochloric acid. The addition of CA extract cause current density decreased. The inhibition of both anodic and cathodic reactions is increasingly pronounced when increasing CAextract concentration. Inhibitors indicated anodic or cathodic type when corrosion potential shifted more than 85 mV of the corrosion potential absence inhibitor[28]. Ecorr shifted less than 85 mV as shown in Table 1 (polarization). It can be classified as the mixed type corrosion inhibitor. The Tafel plots show that the presence of the extract caused a decrease in both the anodic and cathodic current densities.CA. The addition of CAextract to the HCl solution reduces the anodic dissolution of iron and also retards the cathodic hydrogen evolution reactions as would be expected. Both corrosion current density and corrosion rate were considerably reduced in the presence of the extract. These results are indicative of the adsorption of inhibitor molecules on 304SS surface [29]. The cathodic (βc) and anodic Tafel slopes (βa) did not significantly change with the addition of the CA extract. Irregularity of βa and βc showed that the CA extract was a mixed inhibitors modify the mechanism of anodic dissolution and cathodic hydrogen evolution [30, 31]. The highest inhibitor efficiency (99.95%) was obtained by adding 4 g/LCA of extract.

3.3. Temperature effect and activation energy

In order to study the effect of temperature 304SS surface of the inhibitor in 1 M HCl solution, polarization potentiodynamic were studied in the temperature range of 298-328 K in the absence and presence of CA extract shown in Fig. 2.

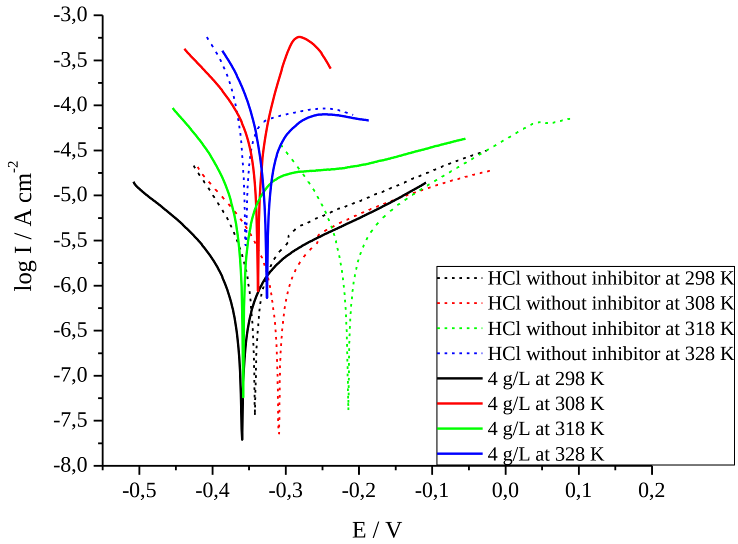

Fig. 2. Polarization curve of 304SS in the hydrocloric acid 1 M absence and presence inhibitor at varied temperature.

Table 2 shows efficiency inhibition deacreases with increasing temperature. It may be due to that the higher temperature maight cause the desorption of the CA extract from the steel surface [32]. IE decrease with increasing temperature caused physisorption on metal surfaces [33]. Physisorption showed the ability of dipoles interactions is relatively weak and at high temperature could cause desorption of BWP extract from the metal surface [34].

The effect of temperature towards the corrosion rate can be declared as Arrhenius equation, Eq. 2. Temperature is essential parameter n studies on metal dissolution. The temperature can modify the interaction between the steel electrode and the acidic media [2]. The value of E_a can be obtained from the slope of linier regression result between lnI_{corr} and 1/T (Fig. 3). The calculation result of $E_a$value with inhibitor is bigger than without inhibitor (Table 3). The increase of

$E_a$value with inhbitor shows that reaction occurs slowly and its corrosion rate is sensitive to temperature [35]. High value of Ea will lower the corrosion rate or lower the corrosion current density. This indicates that the electron transfer in oxidation-reduction process will become less dense and hence it will lower the corrosion rate [13].

Table 2.The potentiodynamic polarization data of 304 SS in the hydrocloric acid 1M absence and presence inhibitor at temperature variations.

Inhibitor (g/L)	*T* (K)	β_a (V/dec)	β_c (V/dec)	E_{corr} (V)	I_{corr} (A/cm^2)	*IE%*
0	298	0.043	0.078	-0.33	$9.83x10^{-7}$	-
	308	0.079	0.137	-0.31	$1.60x10^{-6}$	-
	318	0.095	0.43	-0.21	$5.29x10^{-6}$	-
	328	0.064	-0.21	-0.38	$6.34x10^{-4}$	-
4	298	0.032	0.036	-0.37	$2.29x10^{-8}$	97.67
	308	0.081	0.039	-0.34	$4.11x10^{-7}$	74.31
	318	0.046	0.079	-0.35	$3.67x10^{-6}$	30.62
	328	0.074	0.413	-0.37	$5.56x10^{-4}$	12.30

Arrhenius equation : $\ln i_{corr} = \ln A - \frac{E_a}{R}\left(\frac{1}{T}\right)$ (2)

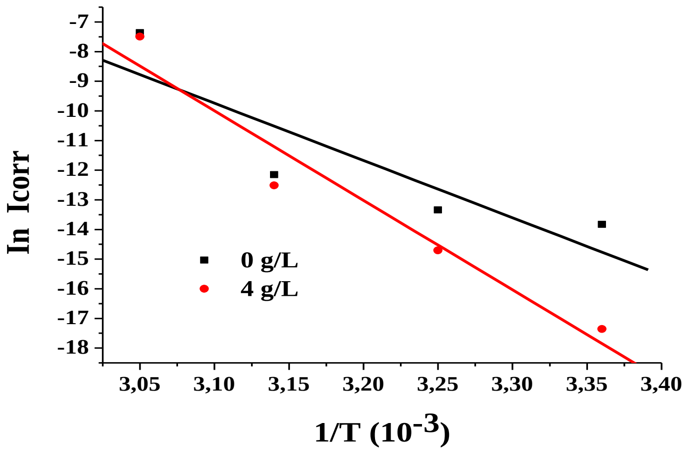

Fig. 3. Arrhennius plot of 304SS in 1M M HClwith and without inhibitor.

Table 3. Activation energy value.

Concentration of extract CA (g/L)	E_a (kJ/mol)
0	161.83
4	246.80

3.4. Adsorption isotherm and thermodynamic calculation

In order to know the mechanism of adsorption, the isothermal adsorption is calculated. Beside the calulation of the isothermal adsorption, thermodinamical calculation must be conducted to gain supporting data of spontaneous corrosion, entaly and entropy as the influence of CA extract addition to the solution.

3.4.1. Adsorption isotherm of CA extract

The adsorption behaviour of the inhibitor on the metal surface reveal the mechanism of corrosion inhibition. In this study, the data were tested graphically by fitting to various adsorption isotherms including the Freundlich, Temkin, Florry-Huggins, Langmuir, ang Frumkin Isotherms. The best fit isotherm followed Frumkin isothherm, Eq. 3. The value of Ea is presented in Table 3 and Fig. 4. These results indicate that the adsorption isotherm of CA extracts CA physisorption which according to the potentiodynamic polarization data.

$$\log\left\{\frac{\theta}{(1-\theta)C}\right\} = \log K_{ads} + a\theta \tag{3}$$

where, C is the concentration of inhibitor, θ is the degree of surface coverage, K_{ads} is the adsorption equilibrium constant and a is an interaction parameter. The value of a takes into account and means the attraction ($a > 0$) or repulsion ($a < 0$) between adsorbed species. When $a = 0$ indicate no interaction and this isotherm becomes equivalent to Langmuir isotherm.

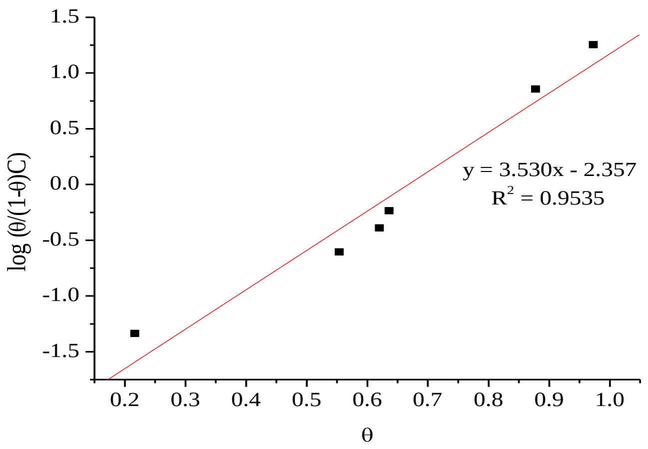

Fig. 4. Frumkin isothermal adsorption.

3.4.2. Thermodynamic calculation

The value of K_{ads} is related to the free energy of adsorption by the Eq. (4).

$$K_{ads} = \frac{1}{55.5} exp\left(\frac{-\Delta G^{\circ}_{ads}}{RT}\right) \tag{4}$$

where, ΔG°_{ads} is standard free energy of adsorption and the value of 55.5 is the concentration of water in solution expressed in mol. The calculated ΔG°_{ads} are given in Table 4. Generally, values of ΔG°_{ads} up to -20 kJ/mol are consistent with electrostatic interaction between charged molecules and a charged metal (physisorption). While those around -40 kJ/mol involves charge sharing or transfer from the inhibitor molecules to the metal surface to form a co-ordinate type of bond (chemisorption) [36]. The ΔG°_{ads} value in this study (Table 4) indicates that CAextract adsorbed via physisorption on the 304SS surface both in 1 M HCl solution. The negative values for ΔG°_{ads}indicate spontaneus adsorption of CAextract [37]. This result support that adsorption of CA extract is physisorption and that in a good agreement with the explanation in section 3.2.

The change of entalpy (ΔH°) andentropy (ΔS°) of activation calculated by the *slope* and *intercept* in linier plot shown in Fig. 3 and Eq. 5 [38].

$$I_{corr} = \left(\frac{RT}{Nh}\right) exp\left(\frac{\Delta S^{\circ}}{R}\right) exp\left(\frac{-\Delta H^{\circ}}{R}\right) \quad (5)$$

where, *h*is Planck's constant, N is the Avogadro's number, T is absolute temperature, and R is the universal gas constant. The $\Delta H°$value and $\Delta S°$ value is presented in Table 4. The positive values of $\Delta H°$both in with and without CA extract reflect the endothermic nature of steel dissolution [32, 37]. The possitive value of ΔS°showed that adsorption process followed with an increase ΔS° , which is the driving force for adsorption inhibitor to the metal surface [38].

Table 4. Thermodynamic parameters for 304 SS in 1M HCl with and without inhibitor.

Inhibitor (g/L)	ΔG°_{ads} (kJ/mol)	$\Delta H°$ (kJ/mol)	$\Delta S°$ (J/mol.K)
0	-	159.01	196.28
4	-12.26	243.87	456.47

3.5. Surface morphology

Surface analysis using scanning electron microscope (SEM) proved a significant improvement on surface morphology of 304SS plates in the addition of CA extract. Figure 5 shows surface morphology of 304SS in HCl with and without inhibitor.

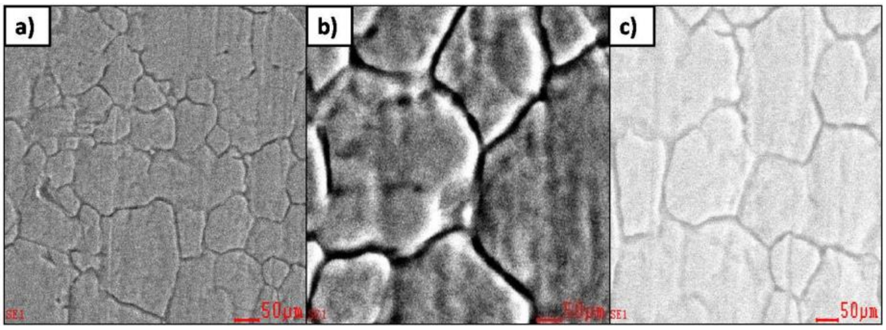

Fig. 5. Micro photograph obtained by SEM for surface morphology of 304SS before corrosion (a), after corrosion in 1 M HClwithout inhibitor (b) and after corrosion in 1 M HCl withinhibitor (c).

The SEM provides means of surface morphology. Fig. 5(a) shows clearly in the surface before immersion smooth and uniform. When sample was immersed in HCl, corrosion pits were created on the surface due to aggressive attack of acid Fig. 5(b). It was indicates deposition of corrosion products. The addition of inhibitor retarded the corrosion rate and formed smooth layer Fig. 5(c). Brigthness of the image indicates good interaction of inhibitor with 304SS surface.

3.6. Inhibition mechanism

The corrosion of 304SS in HCl solution retarded with addition on inhibitor. Based on the polarization measurement, the decrease of IE percentage with the increase of temperature indicates to occur physical adsorption by inhibitor at 304SS surface. Polarization potentiodynamic measurements showed the CA extract retarded corrosion rate which corresponds to molecular adsorption of inhibitor. These organic compound protonated in HCL solution which favored adsorption of these molecules over surface of negatively charged 304SS(electrostatic bonding). In this research adsorbed inhibitor molecules accumulated at the metal/ acid interface and constructed layer surrounding 304SS.

4. Conclusion

CA extractact as good inhibitor on 304SS in 1M HCl. It can inhibit corrosion process. The value of inhibition efficiency obtained is 99.96% based on data of potentiodynamic polarization. Efficiency inhibition deacrease with increasing temperature. Adsorption mechanism which occurs is physisorption term by relying on the Frumkin isotherm equation.

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