International Journal of Engineering

Journal Homepage: www.ije.ir

Exploration of *Eucheuma* Seaweed Algae Extract as a Novel Green Corrosion Inhibitor for API 5L Carbon Steel in Hydrochlorid Acid Medium

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P A P E R I N F O

Paper history:
Received 28 September 2021
Received in revised form 03 December 2021
Accepted 23 December 2021

Keywords:
Eucheuma Extract
Corrosion Inhibitor
API 5L
Electrochemical Measurement
Hydrochloric Acid

A B S T R A C T

Regarding the issue of green chemistry and the vision of human sustainability, a novel corrosion inhibitor for API 5L carbon steel was explored from *Eucheuma* seaweed algae. The inhibiting performance of *Eucheuma* extract was studied by electrochemical measurements such as potentiodynamic polarization and electrochemical impedance spectroscopy (EIS). In this work, the Fourier Transform Infra-Red (FTIR) was also employed to confirm the main phenolic compounds of *Eucheuma* extract. The electrochemical measurement result indicated that the *Eucheuma* extract was an efficient corrosion inhibitor in reducing corrosion attacks of API 5L carbon steel in hydrochloric acid medium. The improved corrosion resistance is attributed to the complicated items, including extract concentration and holding time. *Eucheuma* inhibitor efficiency up to 90% (96.4%) with concentration 500ppm and 30min holding time indicated that *Eucheuma* could be used as a green inhibitor. It was found that the *Eucheuma* extract was a mixed-type corrosion inhibitor that inhibit both the cathodic and anodic corrosion reaction. This study was helpful to discover the seaweed algae that was abundant in Indonesia's marine for inhibiting corrosion of API 5L carbon steel in an aggressive environment.

doi:*10.5829/ije.2022.35.06c.13*

NOMENCLATURE

CR	Corrosion rate, mils per year (mpy)	R_{ct}	Charge transfer resistance (Ω)
EIS	Electrochemical Impedance Spectroscopy	R_s	Solution resistance (Ω)
FTIR	Fourier Transform Infra Red	I_{corr}	Corrosion current density (A/m^2)
OCP	Open Circuit Potential	E_{corr}	Corrosion potential (V)

1. INTRODUCTION

Low carbon steel such as API 5L is one of the most widely materials used and extensively applied for oil and gas exploration, drilling, and production [1]. These steels are important due to their low cost, easily fabrication, excellent welding, and forming abilities. However, they are vulnerable to corrosion attack when exposed to corrosive agents [2].

Hydrochloric acid (HCl) is one of the corrosive agents commonly used in industries for acid cleaning, descaling, and pickling. Corrosion inhibitors can be useful for avoiding the corrosion caused by hydrochloric acid [3].

In the last few years, the use of natural and organic extract as green corrosion inhibitors has attracted many researchers' attention. The green corrosion inhibitors have many advantages such as biodegradable, non-toxic, environmentally friendly and ecologically acceptable, inexpensive, readily available, renewable and safe to use compared to the chemical ones [4].

There are several previous works about environmentally-friendly corrosion inhibitors in a hydrochloric acid medium, such as reviewed by Yang ang Yang [5] and research conducted by Jiddawi et

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Please cite this article as: A. Nikitasari, G. Priyotomo, A. Royani, S. Sundjono, Exploration of *Eucheuma* Seaweed Algae Extract as a Novel Green Corrosion Inhibitor for API 5L Carbon Steel in Hydrochlorid Acid Medium, ***International Journal of Engineering, Transactions C: Aspects*** Vol. 35, No. 03, (2022) 596-603

al. [6] that used organic compound aniline and phenol. Besides that, some researchers used natural sources Rosmarinus officinalis plant [7], Hyalomma insect [8], and the waste from sunflower seedhull [3] as corrosion inhibitors. In contribution to the research on the natural compound for effective corrosion inhibition in HCl medium, this present work uses *Eucheuma* seaweed algae as the new resource.

Eucheuma is a group of red seaweed algae usually found in Indonesia [5]. *Eucheuma* extract has potential to be used as a green corrosion inhibitor due to the exhibits high growth rates and contains high polyphenol [6]. However, this algae has never been studied before as corrosion inhibitors. Due to the purposes below, this research aims to explore of *Eucheuma* seaweed algae extracts to inhibit the corrosive impact on API 5L carbon steel in hydrochloric acid (0.1 M HCl). Potentiodynamic polarization, electrochemical impedance spectroscopy (EIS) and FTIR were used to assess this work.

2. MATERIALS AND METHODS

2. 1. Materials Carbon steel API 5L specimens having a chemical composition in wt % (C, 0.05; Si, 0.13; Mn, 0.73; P, 0.02; S, 0.004; Fe remainder) were used. Carbon steel was cut with a size of 1 cm x 1 cm x 1 cm, connected with cable wire, and mounted with an exposed area of 1 cm^2. Prior the experiment, the specimens were mechanically abraded using different grades (120 – 1200) of silicon carbide emery paper and subsequently cleaned with acetone, then rinsed with distilled water.

2. 2. *Eucheuma* Extract and Medium For the *Eucheuma* seaweed algae extract process, at first, seaweed was cleaned with tap water and dried at room temperature for five days. The dried *Eucheuma* was chopped and collected in 70% of ethanol (maceration) for 72h. The ratio between dried *Eucheuma* and ethanol for the maceration process was 1:3, respectively. The maceration result was filtrated and evaporated to obtain the extract. To investigate the chemical composition of the *Eucheuma* extract, FTIR was employed.

The hydrochloric acid medium that used in this experiment was 0.1 M HCl. *Eucheuma* extract with various concentration i.e. 0, 50, 100, 200, 400, and 500 ppm was mixed into 0.1 M HCl. All specimens were immersed in the medium without and with 30 min of holding time before electrochemical measurement. The temperature of the medium also varied (room temperature, 40^{o}C, and 50^{o}C) to evaluate the influence of temperature on the corrosion rate.

2. 3. Electrochemical Measurement Electrochemical measurements were performed using GAMRY Series G-750 Corrosion Measurement System. For measurement, the three-electrode corrosion cell was used [7]. API 5L specimens with an exposed area of 1 cm^2 were utilized as a working electrode. A platinum and SCE (saturated calomel electrode) electrode were acted as counter and reference electrodes, respectively.

Potentiodynamic polarization was carried out in the potential range of ±250 mV from the OCP potential. The scanning rate of the polarization curve was about 1 mV/s. The electrochemical impedance spectroscopy (EIS) measurements were conducted with the frequency range of 100 kHz and 10 mHz. The measurements were done with a signal of 10 mV peak to peak. In addition, the impedance results were fitted to obtain the charge transfer resistance (R_{ct}). All the electrochemical measurements were recorded in triplicate.

3. RESULT AND DISCUSSION

3. 1. Polarization Test Result Figure 1 illustrates the cathodic and anodic polarization curves for API 5L carbon steel immersed in 0.1 M HCl containing various concentrations of *Eucheuma* green inhibitor at different temperatures without holding time. In addition, potentiodynamic polarization curves for API 5L in 0.1 M HCl solution at various temperatures and concentrations of *Eucheuma* extract with 30 min of holding time are represented in Figure 2. It is observed that by adding inhibitor, the potential tends to be a similar value but current shifts toward less current. Hence, it is indicated that *Eucheuma* extract was a mixed-type corrosion inhibitor and improved the corrosion resistance of API 5L in the hydrochloric acid medium [8]. From Figures 1 and 2, the polarization curve of the specimen without inhibitor at 50^{O}C has the lowest potential value and the highest current. Therefore, the greatest corrosion rate is specimen without inhibitor at 50^{O}C.

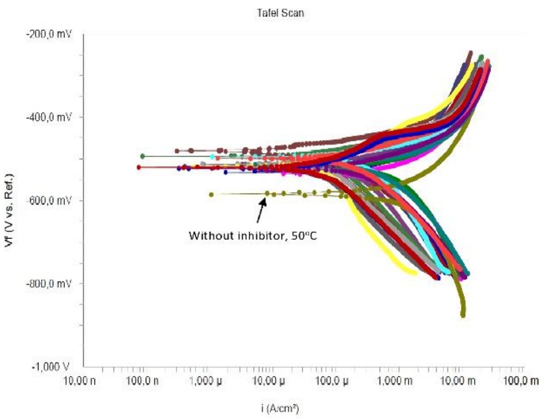


Figure 1. Polarization curves of API 5L in 0.1 M HCl and various *Eucheuma* inhibitor at different temperatures without holding time

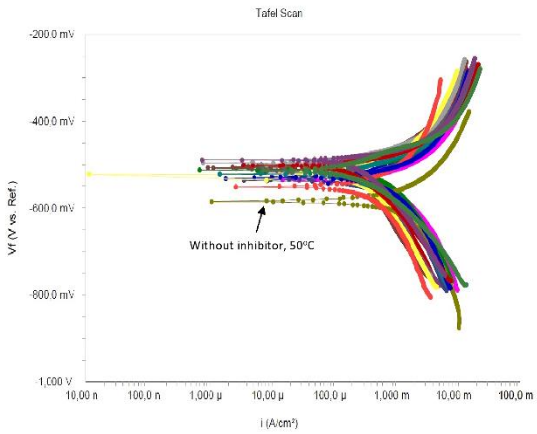


Figure 2. Polarization curves of API 5L in 0.1 M HCl and various *Eucheuma* inhibitor at different temperatures with 30 minute of holding time

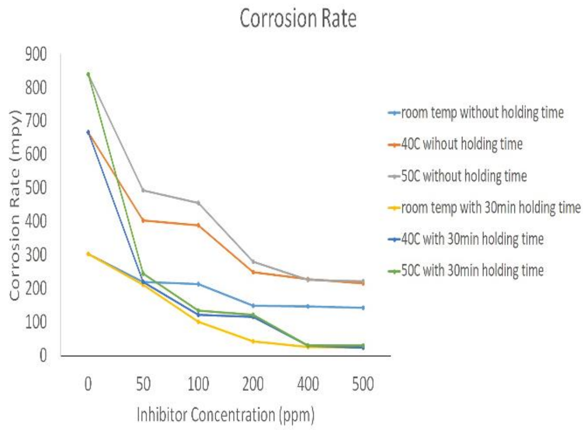


Figure 3. Corrosion rate of API 5L in 0.1 M HCl and various *Eucheuma* inhibitor at different temperatures

In this study, the electrochemical corrosion parameters such as corrosion potential (Ecorr) and corrosion current densities (Icorr) were automatically calculated based on Butler–Volmer and Tafel equations [9]. Tafel slope analysis and fitting of the polarization curves were performed using Echem analyst software to obtain the corrosion rate of API 5L carbon steel. Table 1 listed the parameters of API 5L in 0.1 M HCl and various inhibitor concentrations at room temperature with 30 min of holding time that resulted from polarization test.

According to Table 1, corrosion current density (Icorr) and corrosion rate decrease significantly with increasing inhibitor concentration. Figure 3 summarizes the corrosion rate of API 5L in 0.1 M HCl with various concentrations of *Eucheuma* inhibitor at different temperatures. All specimens with a low inhibitor concentration have a higher corrosion rate than specimens with higher inhibitor concentration. When "30 min holding time" was used, the corrosion rate was lower than "no holding time" specimens. This behavior could justify the adsorption process of inhibitors on the steel surface. The holding time gives a chance to the inhibitor for covering the steel surface. Corrosion rate also boosts with the increasing of temperature based on Figure 3.

TABLE 1. Parameters obtained from polarization test

Concentration (ppm)	Ecorr (mV)	Icorr (mA/cm^2)	Corrosion rate (mpy)
0	-532	6.65E-4	304
50	-523	4.65E-4	212.9
100	-513	2.22E-4	101.5
200	-494	9.58E-5	43.88
400	-481	5.63E-5	25.77
500	-514	5.28E-5	24.17

Inhibition efficiency (η) can be formulated by Equation (1):

$$\eta = \frac{CR - CR(in)}{CR} x100\% \quad (1)$$

where CR and CR(in) are corrosion rate without and with the presence of inhibitor, respectively. Figure 4 showed the inhibitor efficiency of all specimens. Compared to ‘‘no holding time”, specimens with holding time presented a more pronounced inhibition effect.

Inhibitor efficiency increase with ane increas in inhibitor concentration and temperature. The highest inhibition efficiency (96.4%) was specimen containing a *Eucheuma* inhibitor with a concentration of 500ppm with 30 min holding time at 50^{o}C. It is seen that the corrosion rate decreases by adding inhibitor so that it is reduced by 28 times at 500 ppm concentration of the inhibitor. Therefore, it is obvious that the *Eucheuma* extract inhibitor is useful [13].

3. 2. EIS Test Results The inhibition performance of *Eucehuma* extract on API 5L carbon steel was further

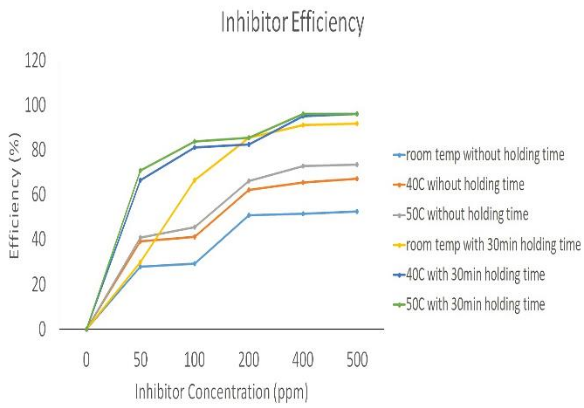


Figure 4. Inhibitor efficiency of API 5L in 0.1 M HCl and various *Eucheuma* inhibitor at different temperatures

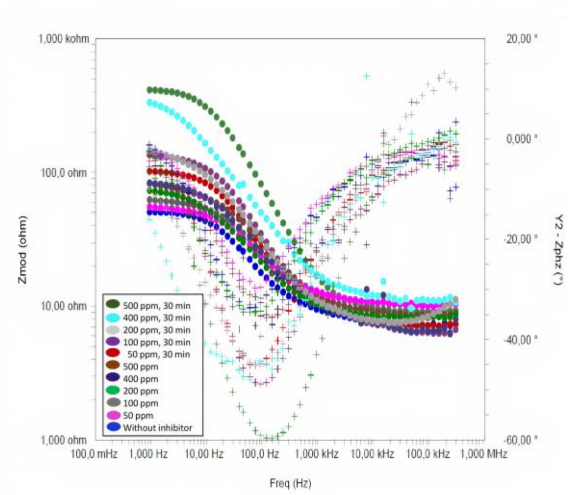
analyzed by means of electrochemical impedance spectroscopy (EIS) measurements under different experimental conditions.

Figure 5 showed a Bode plot for API 5L in 0.1 M HCl environment with and without inhibitor at different temperatures. The peak in the Bode plot indicated the existence of the relaxation time. The higher impedance was obtained for API 5L with the highest concentration of inhibitor in each temperature. Furthermore, the corrosion resistance of steel substrate increased when the concentration of *Eucheuma* extract increased, as the total impedance obviously increased due to the results in Figure 5. According to Figure 5 (a), the phase angles increased from -40 to -60°. The increasing of phase angle with the increasing of inhibitor concentration indicated that the *Eucheuma* inhibitor was physically adsorbed on the surface [14].

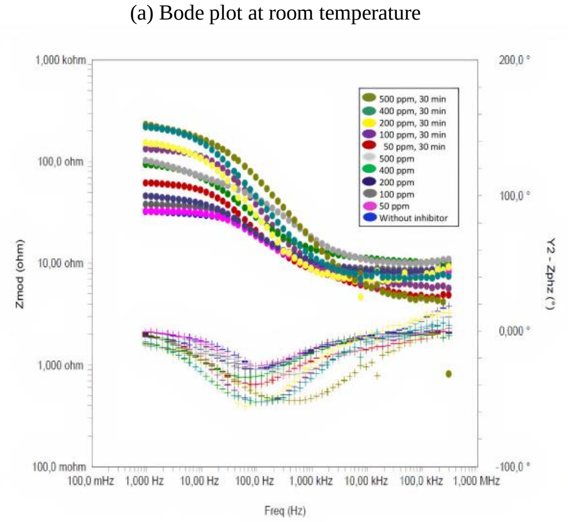
Nyquist plot for API 5L carbon steel in 0.1 M HCl and various inhibitor concentrations with and without holding time at different temperatures presented in Figure 6 Based on Figure 6, all impedance spectra implied a single depressed capacitive semicircle, indicating that the steel dissolution is related to the charge transfer process [15]. In addition, the diameter of the Nyquist plot's semicircle increases with the increasing concentration of *Eucheuma* extract. This phenomenon revealed that *Eucheuma* extract inhibits the corrosion process due to the electronegative charge of the heteroatoms contained in the extracts and the electropositive charge on the steel surface [16].

Electrical circuit model used to interpret EIS data. The suggested electrical circuit model for API 5L in 0.1M HCl and *Eucheuma* inhibitor is shown in Figure 7. The electrical circuit model in Figure 7 includes R_S as solution resistance, R_{ct} as charge transfer resistance, and CPE as constant phase element. In this study, CPE was used instead of capacitance (C) for the circuit model due to all angles of phases are less than 90° based on Figure 5 [10]. CPE was introduced as "capacitance dispersion" that related to capacity of the material surface area of complex surface roughness and inhomogeneous reaction [17]. The CPE element has a fixed phase shift angle and its impedance describes the expression: ZCPE= $1/Y_0(j\omega)n$, where Y_0 and 'a' are the parameters related to the shift phase angle [18].

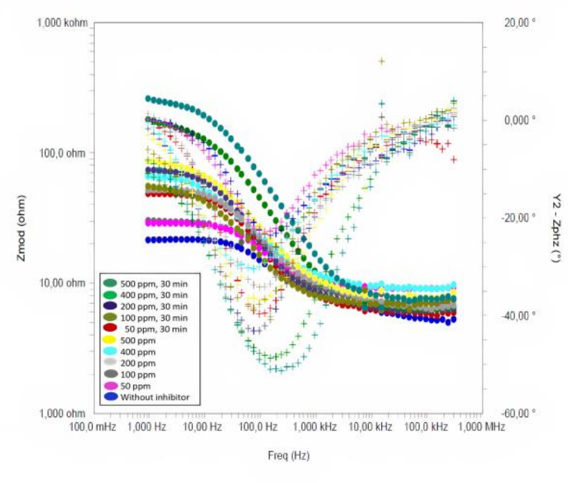
Figure 8 depicts the charge transfer resistance value obtained using the fitting of the electrical circuit model. Charge transfer resistance (R_{ct}) value increase with the increase of inhibitor concentration, as shown in Figure 8. Holding time also increases the R_{ct} value. The increasing R_{ct} value indicates that charge transfer from solution to the steel surface and vice versa inhibited by more inhibitor concentration [17].



(a) Bode plot at room temperature

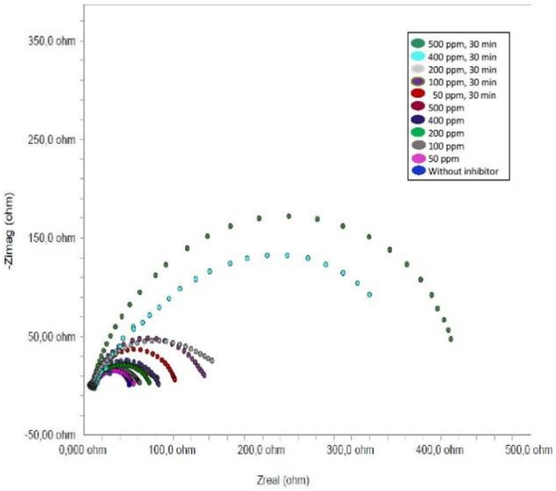


(b) Bode plot at 40ºC

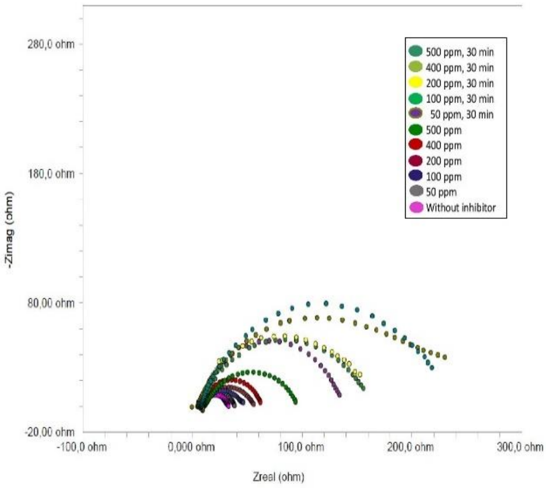


(c) Bode plot at 50ºC

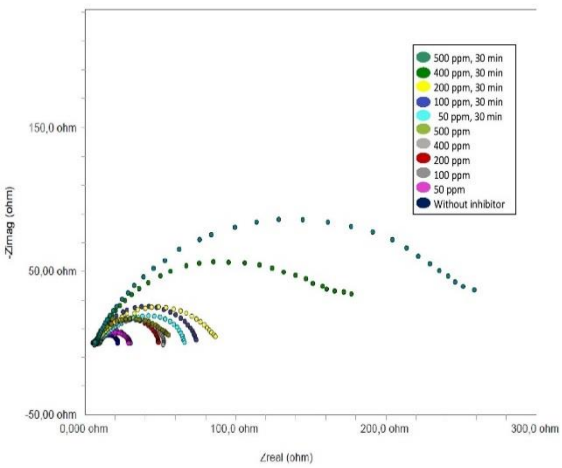
Figure 5. Bode plot of API 5L in 0.1 M HCl and various *Eucheuma* inhibitor concentration



(a) Nyquist plot at room temperature



(b) Nyquist plot at 40ºC



(c) Nyquist plot at 50ºC

Figure 6. Nyquist plot of API 5L in 0.1 M HCl and various *Eucheuma* inhibitor concentration

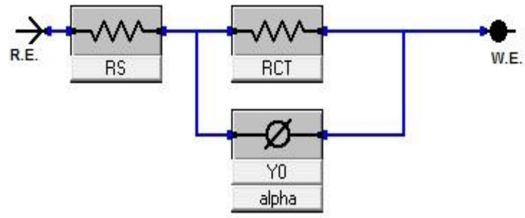


Figure 7. Electrical circuit model

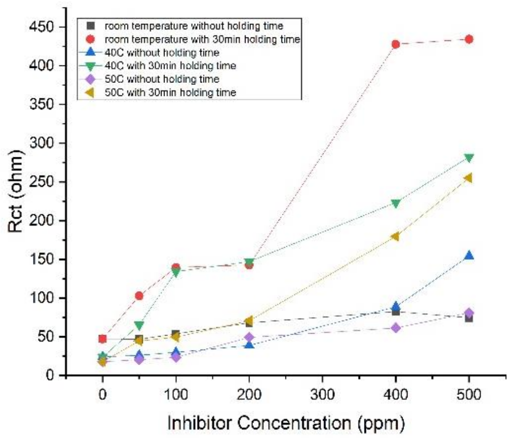


Figure 8. R_{ct} value

3. 3. Thermodynamics of the Electrochemical Process The thermodynamics result of the electrochemical process at various temperature and concentration are listed on the Table 2. The 500 ppm *Eucheuma* inhibitor at 50ºC shows superior inhibition as it has the greatest surface coverage area (0.964).

It has been reported that organic inhibitors are absorbed on the metal surface and adsorption are fitted with Langmuir adsorption isotherms and prevents corrosion. The fundamental details of *Eucheuma* inhibitor also were explored using Langmuir adsorption isotherm, according to the following equation:

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \quad (2)$$

where θ equals to η/100, K_{ads} is the equilibrium constant of the adsorption process, and C_{inh} represents to the inhibitor concentration. Figure 9 depicts plot C_{inh} vs C_{inh}/θ based on Equation (2). The intercept of this plot represents the K_{ads} value. Figure 9 reveals that *Eucheuma* inhibitor follows the Langmuir adsorption inhibitor because the high value of R^2. Therefore, it can be described that the molecules of *Eucheuma* inhibitor are absorbed in a single-layer on the metal surface.

TABLE 2. Thermodynamic parameters

T (K)	Cinh (ppm)	θ	Kads (L/mol)	ΔG (kJ/mol)	ΔH (kJ/mol)	ΔS (J/mol)
298	100	0.666	0.023	-24.84	14.82	133
	200	0.856				
	400	0.915				
	500	0.920				
313	100	0.815	0.029	-26.74		
	200	0.827				
	400	0.954				
	500	0.962				
323	100	0.839	0.036	-28.19		
	200	0.855				
	400	0.963				
	500	0.964				

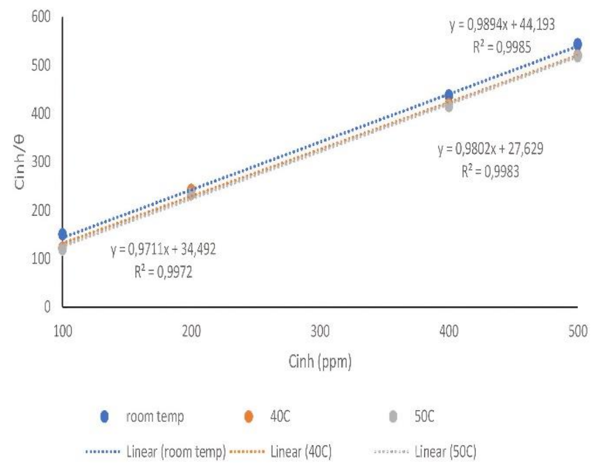


Figure 9. Langmuir adsorption isotherm plot of *Eucheuma* inhibitor at various temperature

Based on Table 2, the maximum value of K_{ads} at temperature 50°C. It indicates the greater adsorbed inhibitors on the metal surface when the temperature increased. Furthermore, the type of adsorption can be acquired with the calculation of adsorption free energy (ΔG). Equation (3) shows the relationship between ΔG and K_{ads}. The value of R is 8.314 J/K. mole and T is absolute temperature in K. Clarifying the calculated result of ΔG, the range of ΔG is -25 kJ/mol and -28 kJ/mol. The negative value of ΔG confirms the feasibility of adsorption process. Moreover, it concluded that inhibition mechanism of the *Eucheuma* inhibitor is mainly physical adsorption on the metal surface and *Eucheuma* inhibitor is chemical adsorption at higer temperature [19].

$$\Delta G = -R.T \ln (10^6 . K_{ads}) \quad (3)$$

Equation (4) is used to determine the value of the heat of adsorption (ΔH) and the entropy (ΔS).

$$\ln K_{ads} = ln\frac{1}{C_{solvent}} - \frac{\Delta H}{R.T} + \frac{\Delta S}{R} \quad (4)$$

ΔH and ΔS are slope and intercept of ln K_{ads} vs T^{-1} curve respectively. These two parameters are presented in Table 2. The positive value of ΔH indicates an increase in irregularities due to desorption of H_2O molecules from surface and adsorption of the inhibitors molecules in each site [3]. The The positive amount of entropy elicits that the thermal stability of the film increases at elevated temperature and their irreversibility process [20].

3. 4. FTIR Test Results

The FTIR of the *Eucheuma* extract is represented in Figure 10. A broad peak with the wavenumber value of 3372.68 cm^{-1} has corresponded to OH bonds. Besides, the strong peak of 2922.28 and 2856.7 cm^{-1} are related to the binding of C-H group. It is also clear that the peaks at 1729.26 and 1631.85 cm^{-1} are related to the vibrational mode of the bindings of C=O and C=C, respectively. The wavenumber values of 1452.46 and 1412.92 cm^{-1} are depicted the bond of O-H. The peak at 1167.95 and 1049.32 cm^{-1} belonged to C-O.

Consequently, it can be concluded that *Eucheuma* extract contained elements of O, H, and C with a cyclic group of carbon or polyphenol compound which attracted the steel substrates according to have free electrons. This behavior could justify the decrease in the corrosion rate of API 5L substrate in HCl solution in the presence of *Eucheuma* extract as a green inhibitor.

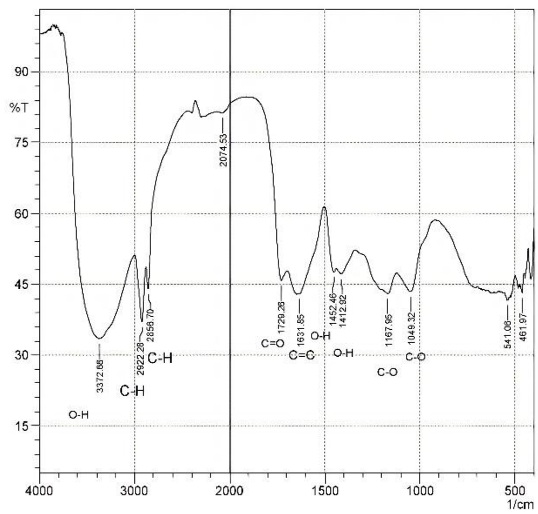


Figure 10. FTIR result of *Eucheuma* extract

4. CONCLUSION

Eucheuma seaweed algae was successfully used for the first time as a green corrosion inhibitor for API 5L carbon steel in the hydrochloric acid medium. Corrosion resistance of API 5L boosts with the concentration of *Eucheuma* inhibitor and holding time. Temperature also improves efficiency of inhibitor. In the presence of 500 ppm inhibitor with 30 min holding time at 50ºC, the inhibition efficiency reaches about 96.4%. The corrosion rate at this concentration of inhibitor was also reduced by about 28 times.

The charge and ion transfer on the steel surface decrease because of the complex formation between the inhibitor and the ions of the steel surface. Therefore, the charge transfer resistance will improve and the corrosion rate will reduce with the existance of *Eucheuma* inhibitor. The inhibiton mechanism of *Eucheuma* inhibitor follows Langmuir adsorption isotherm. *Eucheuma* inhibitor is adsorbed on the metal surface forming a passive layer that can prevent corrosion process. These results confirm that the introduction of aromatic rings and OH to organic compounds is suitable as a corrosion inhibitor in the hydrochloric acid medium.

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Persian Abstract

چکیده

با توجه به موضوع شیمی سبز و چشم انداز پایداری انسان، یک بازدارنده خوردگی جدید برای فولاد کربنی API 5L از جلبک جلبک دریایی *Eucheuma* مورد بررسی قرار گرفت. عملکرد بازدارندگی عصاره *Eucheuma* با اندازه‌گیری‌های الکتروشیمیایی مانند قطبش پتانسیودینامیک و طیف‌سنجی امپدانس الکتروشیمیایی (EIS) مورد بررسی قرار گرفت. در این کار، تبدیل فوریه مادون قرمز (FTIR) نیز برای تایید ترکیبات فنلی اصلی عصاره ی *Eucheuma* به کار گرفته شد. نتایج اندازه گیری الکتروشیمیایی نشان داد که عصاره *Eucheuma* یک بازدارنده خوردگی کارآمد در کاهش حملات خوردگی فولاد کربنی API 5L در محیط اسید هیدروکلریک است. مقاومت در برابر خوردگی بهبود یافته به موارد پیچیده، از جمله غلظت عصاره و زمان نگهداری نسبت داده می شود. راندمان بازدارنده Eucheuma تا ۹۰٪ (۹۶/٤٪) با غلظت ppm ۵۰۰ و زمان نگهداری ۳۰ دقیقه نشان داد که عصاره *Eucheuma* می‌تواند به عنوان یک مهارکننده سبز مورد استفاده قرار گیرد. مشخص شد که عصاره *Eucheuma* یک بازدارنده خوردگی نوع مخلوط است که هم واکنش خوردگی کاتدی و هم آندی را مهار می کند. این مطالعه برای کشف جلبک دریایی که در دریای اندونزی برای مهار خوردگی فولاد کربنی API 5L در یک محیط تهاجمی فراوان بود، مفید بود.