Effectiveness of Red Dragon Fruit Peel Extract as an Eco-Friendly Corrosion Inhibitor for API 5L Steel

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Abstract. The development of organic or natural inhibitors is essential due to their cost-effectiveness and eco-friendliness. In this study, an organic inhibitor derived from red dragon fruit peel waste is employed. The material of interest is heat-treated API 5L Grade B steel. The research assesses the inhibitor's efficiency in corrosive environments, namely 3.5‰ NaCl solution and pH 3 HCl solution, with varying inhibitor concentrations (5 ppm, 10 ppm, 15 ppm, 20 ppm). Using Tafel analysis and Electrochemical Impedance Spectroscopy (EIS), it is found that the addition of red dragon fruit peel extract substantially reduces the corrosion rate. The highest efficiency is observed in the 3.5‰ NaCl solution (62.4%) and pH 3 HCl solution (87.73%), accompanied by charge transfer resistances of 2.47 x 106 Ω and 1.79 x 103 Ω, respectively. Immersion tests reveal corrosion products, subsequently examined using X-ray Diffraction (XRD) to identify lepidocrocite phases in the 3.5‰ NaCl solution and magnetite phases in the pH 3 HCl solution. This research supports the development of environmentally friendly and effective natural corrosion inhibitors for protecting steel materials in corrosive conditions.

1 Introduction

Application of the American Petroleum Institute Spec 5L (API 5L-B Steel) is widely used in the mining industry, as a pipeline for gas, fluid, and oil transportation applications as well as distribution of exploration and production processes [1,2]. The biggest problem that occurs in piping systems is related to corrosion due to the influence of the concentration of chloride ions dissolved in the water medium so that the environment is corrosive(3). Corrosion has the potential to cause material failure [4], [5], [6]. Corrosion cannot be stopped, but corrosion can be reduced and inhibited in various ways namely using corrosion inhibitors[7]. Cathodic protection [8], coating [9], and others. Recent developments have shown that the use of inhibitors is the most effective and efficient way to protect corrosion on metals [10]. The use of commercial inhibitors containing synthetic compounds from inorganic elements has proven to be quite effective as metal corrosion inhibitors such as phosphate [11], silicate and phosphonate [12], Molybdate [13], and tungstate [14], but at the same time it requires high costs in management, regarding the nature of dangerous toxins as the implications of their use, forcing researchers to seek alternative corrosion inhibitors, namely organic inhibitors that are environmentally friendly, nontoxic and low cost for metal corrosion protection [15]. Many organic corrosion inhibitors have been developed from natural environmentally friendly materials that utilize plant parts such as leaves, roots, fruits, and stems that contain antioxidant compounds[16]. One plant that contains antioxidants is Red dragon fruit (Hylocereus polyrhizus). Red dragon fruit is a fruit from the Cactaceae tribe, which is widely consumed in Indonesia. Research on the efficacy of red dragon fruit (Hylocereus polyrhizus) has also been widely conducted [16]. However, the extensive and many studies related to the utilization of Red dragon fruit is only fixated on the flesh, while the skin has not been used optimally. Even though the red dragon fruit skin contains vitamin C, vitamin E, vitamin A, protein, fat, carbohydrate, crude fiber, flavonoids, polyphenols, and phytoalbumin [17]. The advantage of Red dragon fruit skin, which is rich in polyphenols, flavones, flavonoids and is a source of antioxidants. This is in accordance with the research of Nurliyana, et.al. [17] which states that the antioxidant activity of the red dragon fruit skin is greater than the antioxidant activity in the flesh, so that it has the potential to be developed into a source of natural antioxidants. Compound components these organics usually have functional groups composed of N, S, or O atoms and functional groups and double bonds in the molecule which facilitate adsorption on the metal surface [18]. The inhibition efficiency of organic compounds is related to the adsorption properties which will then be able to form complex compounds with metals, so that the metal becomes protected [19].

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Corrosion inhibitors mostly come from flavones, flavonoids [20], so that the skin of Hylocereus polyrhizus extract can potetially be an organic inhibitor that can protect the steel surface from corrosion

In this work, the effectiveness of Red dragon fruit (Hylocereus polyrhizus) skin extract in the corrosion inhibition of API 5L Grade B steel in the corrosive enviroment of NaCl and HCl solutions is systematically presented. The rate of corrosion is measured using the Tafel extrapolation method and electrochemical impedance spectroscopy (EIS), The corrosion rate and resistance of the test specimens were tested using the weight loss method, while the crystallization phases and peak intensity on the corrosion test specimens were tested using XRD (X-ray Diffraction). This research is expected to make a beneficial contribution to the study of corrosion inhibition by organic substances; with the possible future introduction of inhibitor trading schemes, it could be used as an alternative means of corrosion inhibition in steel pipes, which are mostly used in the oil and natural gas industries.

2 Material and Methods

2.1 Sample Preparation

The material specimen API 5-L grade B contains elements such as 0.148% C, 0.198% Si, 0.004% S, 0.012% P, 0.425% Mn, 0.010% Ni, 0.008% Cr, <0.005% Mo, <0.002% Ti, <0.007% Cu, <0.002% Nb, <0.002V, <0.002% Al. The specimens is prepared in a size of 1.5 cm x 1.5 cm, then in a circle with a size of 1 cm^2. The test sample was mirror with emery papers of grade 120 to 800 consecutively, cleaned with distilled water, and dried in air.

2.2 Inhibitor Preparation

Red dragon fruit skin cleaned with water, mashed and soaked in 70 % ethanol for 48 hours. Filtering is carried out to obtain maceration results. The maceration results are evaporated with a rotary evaporator at a temperature of 50ºC. Inhibitor solution is made by mixing distilled Aquades and red dragon fruit skin powder and thus becomes a solid extract

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2.3 Solutions Preparation

The main solution of corrosive media, NaCl 3.5%, This solution is made from distilled water mixed with technical NaCl salt, for the volume of 1 liter of distilled water, 35 grams of technical NaCl can be added to produce 3.5% NaCl and HCl with pH=3 with the addition of variations of inhibitors 0 ppm, 5 ppm, 10 ppm, 15 ppm, and 20 ppm.

2.4 Fourier Transform Infra-Red Spectroscopy

Fourier Transform Infra-Red (FTIR) spectroscopy has been developed for analysis characteristics of the Functional group in the inhibitor extract from Skin of Red dragon fruit (Hylocereus polyrhizus). In testing using FTIR Spectroscopy, the light source is medium infrared, the detector used is Deuterated Lanthanum Triglycine Sulfate (DLATGS). Sample preparation used a basting technique which in involved inhibitor extract Hylocereus polyrhizus sample in a window of 1 drop (0.2 ml) until it can be penetrated by light. The spectrum of the propylene glycol solvent that functions as a background was stored as a reference. FTIR tool operations were operated until a spectrum was obtained from the sample holder. In addition to the sample analyzed also the spectrum of extract from Skin of Red dragon fruit as a comparison. The infra-red Spectrum in OPUS format was stored in a data point table format that can be opened using Microsoft Excel software. The spectrum data used was the overall absorption data

2.5 Weight loss and X-ray diffraction (XRD)

The weight loss test Using the weight loss method according to ASTM G31-72 [21] used a specimen with a diameter of 30 mm, cleaned according to ASTM G-1-90 [22] Analysis on the weight loss test is carried out on the measurement of the initial weight and weight after testing the specimen. To determine the corrosion rate by using the weight loss method, the following equation is used

$$\mathbf{Cr} = \frac{k.\ Wg}{\delta\ A\ T} \quad (1)$$

Where Cr is the corrosion rate weight loss test (mpy), k= 87.6 is the constant, Wg is the ratio between the initial weight measurement and the weight after test, δ is the density of the specimen (g/cm^3), A is the cross-sectional area of the specimen (cm^2) and T is the exposure time (hour). After weight loss testing. Corrosion products on the corrosive medium of NaCl and HCl solutions were then immediately tested using XRD (X-ray diffraction) testing to determine the crystallization phase of the corrosion products.

2.6 Electrochemical Measurements

The electrochemical test used the Tafel polarization method and EIS. Electrochemical testing using a potentiostat test apparatus according to ASTM G-87. were used to determine corrosion rate. The electrochemical cell consisted of a conventional three-electrode configuration with graphite as the counter electrode and a saturated calomel electrode (SCE). In the potentiostat test there is a reference electrode which functions to measure the potential value and a platinum cable that functions as a counter electrode, and the working electrode is a steel specimen API 5L Grade B. Tafel polarization test uses 250 mL of test solution containing a variety of red dragon fruit peel extract inhibitors in a solution of NaCl 3.5 % and HCl pH-3. In the Tafel polarization test, the polarization curves performed with a scan rate of 0.2 mVs^{-1} in the potential range of ± 250 mV proportional to the potential of corrosion. All the potentials recorded with respect to the SCE. Data analysis using software Gamry 6.25 repetition is carried out for each test in open room conditions and at room temperature. Prior to measurement, the specimen was immersed in a corrosion medium at open circuit potential until it reached steady state conditions. Corrosion current density (Icorr) is determined from the intercept of extrapolated cathodic and anodic Tafel polarization slopes. The corrosion rate (Cr) can be calculated using the formula

$$\mathbf{Cr\ (mpy) = 0.129\ \frac{Icorr.M}{\delta}} \quad (2)$$

Where M(g/mol) is the equivalent weight, δ is the density of the specimen (g/cm^3). Meanwhile, the inhibition efficiency (EI) used as a measure of the effectiveness of the inhibitor at each addition of inhibitor concentration is obtained by the following equation

$$\mathbf{EI\ \% \ = \frac{Icorr - Icorr_{inh}}{Icorr}\ x\ 100\ \%} \quad (3)$$

Where Icorr is the corrosion current density without an inhibitor and $\mathrm{Icorr_{inh}}$ is the corrosion current density with the addition of the inhibitor concentration. The EIS measurements are used to determine electrochemical kinetics parameters is related to electrical elements such as resistance, capacitance and inductance after adding the inhibitor [23]. Measurements were made of the charge transfer resistance and electrical interface of the specimens at the corrosion potential (Ecorr) in the frequency range 10.000 Hz to 10 MHz with an amplitude of 10 mV. The resistance efficiency (EI), is calculated using the equation

$$\mathbf{EI\ \% \ = \frac{RC_I - RC}{RC_I}\ x\ 100\ \%} \quad (4)$$

where RC and RC_I referred to as the charge transfer resistance without and with the addition of the inhibitor, respectively.

3 Result and Discussion

Fourier Transform Infra-Red (FTIR) spectroscopy has been developed for analysis characteristics of the Functional group in the inhibitor from Skin of Red dragon fruit (Hylocereus polyrhizus) skin extract. Testing the content of compounds by FTIR testing resulted in functional groups in the red dragon fruit peel extract are shown by the spectrum in figure 1, there were functional groups including O-H (acids), C=O, and C-O. This band confirms the presence of a single bond stretch near 3600-3300 cm^{-1} identified as an O-H functional group characterized by its broad and undivided spectra. Detected as alcohols, phenols, broad absorption near 3400-3300 cm^{-1} Confirm this by finding C-O near 1300-1000 cm^{-1} Obtained through literature frequency data [24,25]. The following functional groups produce alcohols, phenols, flavonoids which are usually found in natural inhibitors [26]. The polarization test was carried out by evaluating the cathodic and anodic slopes of Tafel. Polarization testing is carried out by evaluating the cathodic and anodic slopes of Tafel as shown in figures 2(a), 2(b). Where 2(a) the results of the polarization test on the corrosion medium of 3.5% NaCl solution and 2(b) are the results polarization test on HCl solution pH-3. The data obtained on the Tafel polarization curve are corrosion rate (Cr), Ecorr, and Icorr. Symmetry of energy limiting in the anodic and cathodic directions (according to direction), where as a rule of thumb for the type of inhibitor is the Ecorr shift for the entire concentration range less than ± 85 mV [27].

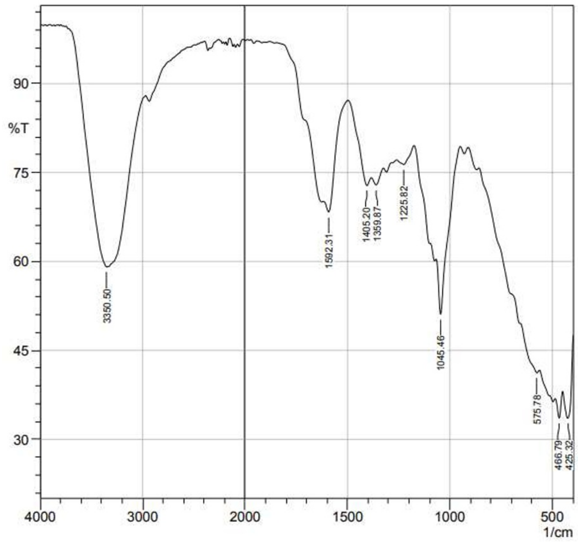

Fig. 1. Characteristics of the Functional group in the Skin of red dragon fruit extract (*Hylocereus polyrhizus*)

The reduction in the corrosion rate was closely related to the addition of inhibitors both on the 3.5% NaCl medium and for the HCl corrosion media pH-3 (**Table 1**). From the data obtained at blank conditions (without inhibitor) the corrosion rate (Cr) is 21.85 mpy on NaCl. and 111.46 mpy on HCl. Then after adding the inhibitor concentration into the corrosion medium, it was indicated that it had an effect on reducing the corrosion rate significantly. The increase in the addition of inhibitor concentration resulted in a decrease in the corrosion rate of API 5L Grade specimens.The highest inhibition efficiency (EI) (equation 3) was found at the inhibitor concentration of 20 ppm where the corrosion rate (Cr) was 9.495 mpy and resulted in an inhibition efficiency of 56.54%. While testing in the HCl pH-3 corrosion medium, the most effective efficiency was at the addition of 20 ppm where the corrosion rate Cr was 12.14 mpy and resulted in an inhibition efficiency (EI) of 89.10%. As described in Figure 3 for the relationship between concentration and inhibition efficiency, that the addition of inhibitor concentration will have the effect of increasing the inhibition efficiency (EI).

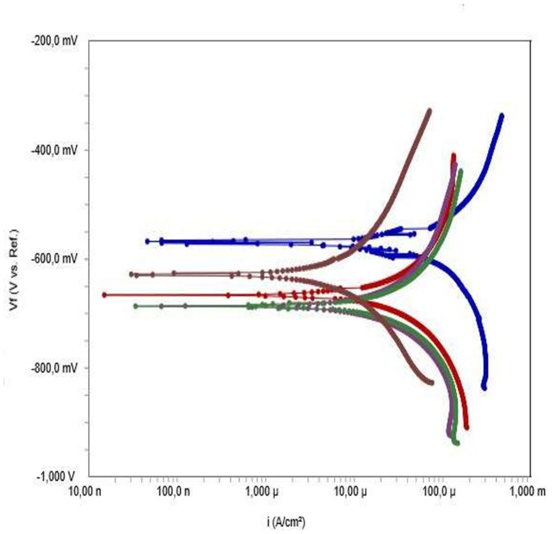

Fig. 2(a). Potentiodynamic polarization curves for API 5L-B steel in NaCl 3.5%, with various concentration Skin of red dragon fruit extract (Hylocereus polyrhizus). Blue circles denote blank, Red circles denote 5 ppm, purple circles denote 10 ppm, green circles denote 15 ppm, and brown circles denote 20 ppm

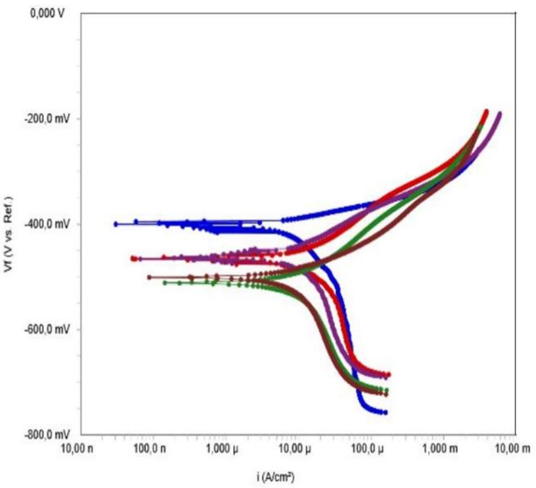

Fig. 2(b). Potentiodynamic polarization curves for API 5L-B steel in HCl pH-3, with various concentration Skin of red dragon fruit extract (Hylocereus polyrhizus). Blue circles denote blank, Red circles denote 5 ppm, purple circles denote 10 ppm, green circles denote 15 ppm, and brown circles denote 20 ppm

Table 1. The corrosion parameters for API 5L-B steel in 3.5% NaCl and HCl pH-3 corrosion medium studied by electrochemical polarizations

Corroson medium	Concentration (ppm)	Icorr (A/cm²)	Corrosion rate	Rct (Ω)	Ru (Ω)	E (%)	Coverage area (θ)
	Blank	24.50	21.85	2.97 x10^{7}	41.25	-	-
	5	21.81	19.54	2.19 x10^{8}	41.65	10.57	0.1057
3.5% NaCl solution	10	15.18	13.60	2.90 x10^{8}	37.80	37.75	0.3775
	15	12.48	11.19	3.40 x10^{8}	40.60	43.93	0.4393
	20	10.50	9.49	3.84 x10^{8}	43.49	56.54	0.5654
HCl pH 3 solution	Blank	124.36	111.46	240.7	311.40	-	-
	5	55.62	50.51	666.5	121.70	54.68	0.5468
	10	50.86	45.58	687.1	38.75	59.10	0.5910
	15	38.18	34.21	749.5	45.99	69.30	0.6930
	20	13.55	12.14	4.18 x10^{3}	1.64x 10^{3}	89.10	0.8910

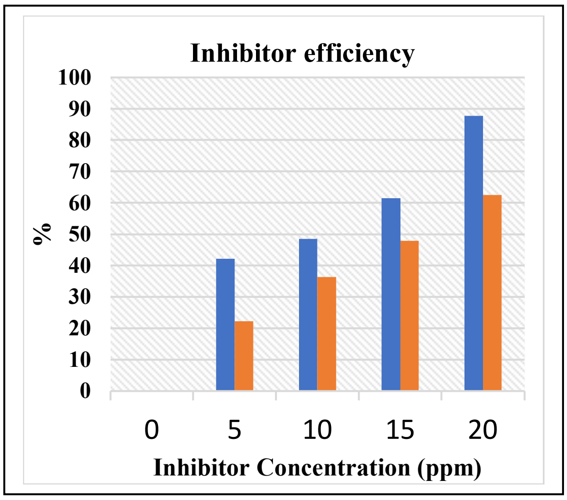

Fig. 3. Typical the relationship between concentration and inhibition efficiency (EI) in corrosive media. Blue lines denote NaCl 3.5% and Red lines denote HCl pH-3

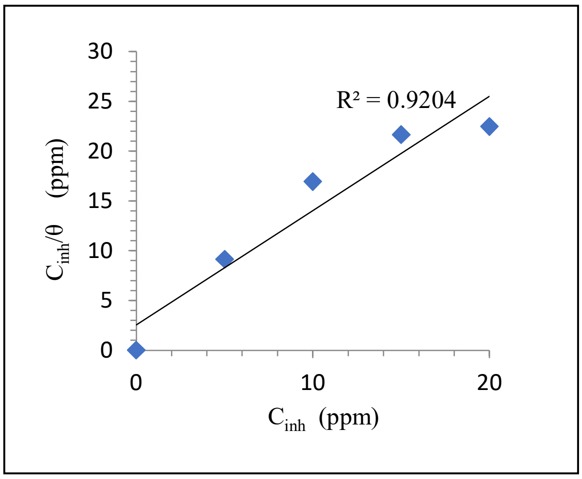

Fig. 4. Langmuir adsorption plots for API 5L-B in HCl pH 3 solution in presence of various concentration of Red dragon fruit (*Hylocereus polyrhizus*) skin extract

This shows that with the addition of an inhibitor to the solution, the resulting current density tends to decrease. A decrease in current density implies to decrease the corrosion rate [27]. The analysis has clearly indicated the corrosion mechanism that occurs due to the addition of IRSE containing anti-oxidants. From the polarization curve, it is known that there is a slowing of the cathodic reaction seen from the Ecorr and Icorr values in the cathodic polarization region. This indicates the inhibition of the cathode reaction on polarization. It is clear that if the oxygen level is reduced in a 3.5% NaCl solution, the corrosion-oxidation-reduction reaction in seawater (reactive NaCl 3.5%) will be inhibited [27,28]. The inhibitor acts as anodic in this experiment, which is distributed due to metal solubility and absorption of the inhibitor at anodic potentials, while the metal specimen is related to the evolution of hydrogen at the cathodic potential [29]. **Table 1** shows that increasing the concentration of the inhibitor significantly reduces the Icorr value. The lowest Icorr value was 10.50 A/cm^2 with a concentration of 20 ppm red dragon fruit peel extract at 3.5% NaCl. Whereas in HCl pH-3 the lowest Icorr was 13.55 A/cm^2 at a concentration of red dragon fruit skin extract of 20 ppm. This pattern clearly shows that the presence of an inhibitor affects anodic and cathodic reactions [30].

The results of polarization testing on the HCl pH-3 corrosion medium at blank conditions obtained the corrosion rate value was 111.46 mpy with the addition of 5 ppm inhibitor concentration, the corrosion rate decreased to 50.51 mpy shown that the corrosion rate decreased steadily with each addition of inhibitor concentration. At 10 ppm the corrosion rate (Cr) is 45.58 mpy. The addition of inhibitor at 15 ppm the result of the corrosion rate was 34.21 mpy. **Table 1** show that the value of the decrease corrosion rate, the largest was in the addition of the inhibitor concentration of 20 ppm which obtained a corrosion rate of 12.14 mpy (resistance efficiency 89.10%). When compared to the inhibition efficiency (EI) of the corrosion media used, it is known that the addition of the inhibitor concentration to the HCl pH-3 corrosion media is more effective in reducing the corrosion rate of API 5L grade B steel material copared to the 3.5% NaCl corrosion media. In general, the increase in inhibition effciency can be due to the increase in surface area coverage of API 5L-B steel by Inhibitor due to blocking sites available for reactions on the surface [31].

The inhibitory mechanism provided by the inhibitor begins with absorption of the inhibitor to the metal surface, then continues with chemical absorption to form a coordination bond with the corrosive solution [32]. In testing using HCl pH-3corrosion medium (**table 1**) demonstrated that the increasing concentration of inhibitor has the potential to form a wider adsorption inhibitor on the metal surface (the highest at the inhibitor concentration of 20 ppm, coverage area (θ) = 0.8910). The relationship between inhibitor concentration and corrosion rate and between concentration and inhibition rate has been studied by Sieverts and Lueg [7] and the description is called an adsorption isotherm. The absorption of organic compounds follows the Langmuir rule of the adsorption isotherm with equation [7]

$$\frac{C_{Inh}}{\theta} = \frac{1}{K_{ads}} + C_{Inh} \qquad (5)$$

Where C_{Inh} Inhibitor Concentration in the electrolyte and K_{ads} the equilibrium constant for the absorption process. Figure 3 shows the isotherm curve of the red dragon fruit peel adsorption inhibitor on the surface of API 5L Grade B steel material in HCl solution. Shown a linear relationship between C_{Inh}/θ and C_{Inh} following Langmuir's rule of the adsorption isotherm (**Eq.5**) using linear regression method. Through the linear regression method R^2 = 0.9204, this shows that a good inhibitor activity and adsorption on the steel surface of API 5L Grade B is in accordance with the Langmuir isotherm. This suggests that the larger the coverage area surrounding the enhanced protection surface will result in increased inhibition efficiency[30]

In the EIS test, the data obtained is the resistance value found in the inhibitor variation. In the test without inhibitor in NaCl corrosion medium, the interface layer resistance (Rct) (**table 1)** value was 2.97 x 107 Ω. The value of resistance for each addition of inhibitor has a steady increase. The greatest resistance value is found in the addition of 20 ppm inhibitor variation which results in a resistance of 3.84 x 108 Ω. In the test medium of HCl corrosion without inhibitor, the resistance value was 240.7 Ω. The value of resistance after being given the addition of an inhibitor has a stable increase where at the addition of 15 ppm the resistance value obtained is 749.5 Ω. The greatest resistance value is found in the addition of a 20ppm inhibitor variation which produces a resistance of 4.18 x 103 Ω. Meanwhile, the HCl pH-3 corrosion media produced corrosion rates of 57.99 and 49.82 mpy respectively.

Alternative testing is also presented on this weightloss method according to ASTM G31-72 [21] as a "Planned Interval Test" ie a procedure to evaluate the effect of time on metal corrosion and also on the corrosive properties of the environment in laboratory testing. As also done by Wachter and Treseder [33] The procedures and results evaluations carried out through "Planned Interval Test". In the weighloss test there is situation data on the corrosion medium NaCl 3.5% corrosive properties of the material. It is know that the corrosion solution medium unchanged and the corrosive properties of the material are decreased where the corrosion solution medium and the corrosive properties in weighloss testing Using the weight loss method according to ASTM G31-72[21] In this method the immersion is carried out on the material test, then calculated the initial weight and after immersion.

The corrosion rate was determined using **equation 1**. As described in table 2 the material codes A1 and B were immersed for 1 week in 3.5% NaCl corrosion medium resulting in a corrosion rate of 18.97 mpy respectively. Meanwhile, the HCl pH-3 corrosion media produced corrosion rates of 63.26 and 53.77 mpy, respectively. Material code A3 were immersed for 3 weeks while A4 were immersed for 4 weeks in corrosion media 3.5% NaCl resulting in corrosion rates of 18.98 and 18.19, respectively. Whereas in the corrosion media HCl pH-3 the corrosion rates of A1, B, A3, A4 were 63.26, 53.77, 57.99 and 49.82 mpy. Test results and literature surveys reveal the presence of the following key elements in the IRSE is carbohydrate, crude fiber, flavonoids, polyphenols, flavones, phytoalbumin and is a source of antioxidants [34] The corrosion inhibition mechanism that occurs is described on the basis of the adsorption process. The constituent structure present in IRSE, Anodic inhibition is possible due to the adsorption of the main phytochemical constituents present in the extract via the oxygen atom/or ring of oxygen atoms in Flavonoids and others. These organic

Table 2. The results data of weight loss testing on corrosion medium of the material are decreased

Corrosion medium	Material code	A	Wo	W1	Cr
NaCl 3.5%	A1	8.302	17.04	16.98	18.97
	B	8.302	17.39	17.33	18.97
	A3	8.302	17.61	17.43	18.98
	A4	8.302	17.30	17.07	18.19
HCl-pH3	A1	8.302	17.35	17.15	63.26
	B	8.302	17.45	17.28	53.77
	A3	8.302	17.44	16.89	57.99
	A4	8.302	17.31	16.68	49.82

4 Conclusion

Red dragon fruit (Hylocereus polyrhizus) skin extract has the potential to be an inhibitor because the functional groups contained in the extract can be categorized as functional groups in organic inhibitors. The maximum inhibition was determined by the potentiodynamic method where the NaCl corrosion medium produced resistance equal to 3.84 x 108 Ω and 4.18 x 103 Ω on the HCl medium. The increase in inhibitor efficiency is directly proportional to the addition of variations in inhibitor concentration. Increasing the concentration of 5 to 20 ppm affects the efficiency of the inhibitor 10.57% to 56.54% in NaCl corrosion medium and 54.68% to 89.10% in HCl corrosion medium. corrosion rate of API 5L Grade B steel in corrosive medium decreases with increasing propolis concentration, where an increase in the concentration of 5 to 20 ppm has an effect on reducing the corrosion rate by 19.54 to 9.495 mpy on NaCl corrosion media and 50.51 to 12.14 mpy on HCl corrosion media. Inhibition mechanism on IRSE by means of adsorption. Where the CInh / θ plot which depends on the extract concentration is a positive gradient which indicates that the adsorption of this extract follows the Langmuir isotherm. IRSE will work optimally and effectively on the HCl corrosion medium because the efficiency level of the HCl corrosion medium is better than the efficiency level in the NaCl corrosion medium.

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