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Article

Corrosion Inhibitor from Sambang Darah Leaves Extract (*Excoecaria cochinchinensis* L.) on Mild Steel in HCl

Vira Anggita Gusniardi, Sanaq Elfira Putri, Suryati, dan Emriadi*

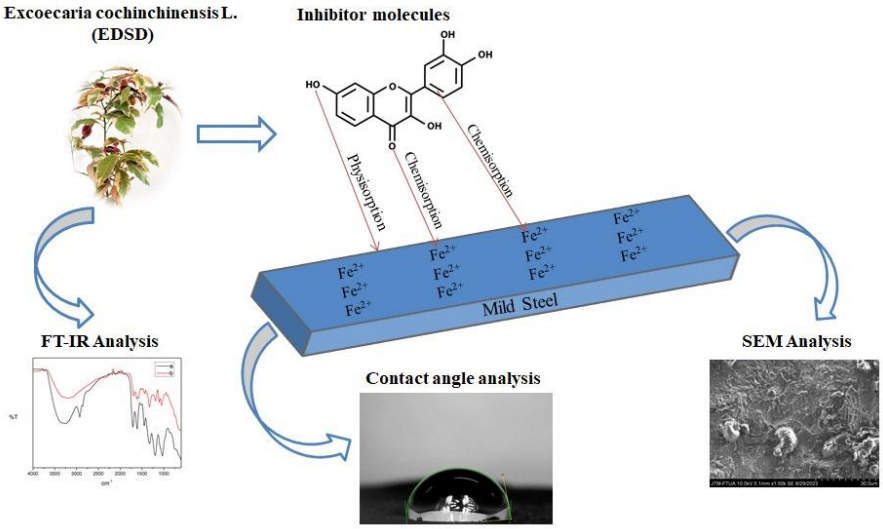
Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Andalas, Padang 25163, West Sumatra, Indonesia

Abstract

Corrosion inhibition studies in mild steels in HCl medium by extracts of sambang darah leaves (*Excoecaria cochinensis* L.) (EDSD) have been conducted using weight loss methods, Fourier-Transform Infrared Spectroscopy (FTIR), contact angle and Scanning Electron Microscopy (SEM). The study is aimed at determining the effectiveness of EDSD inhibition as a corrosion inhibitor in HCl medium. The inhibition efficiency was found to increase with increasing concentration of EDSD and decrease with increasing temperature. The results showed that EDSD was capable of inhibiting mild steel corrosion with an inhibitory efficiency of 87.972% at EDSD 6 g/L in 1 M HCl solution. EDSD adsorption on mild steel surfaces follows Langmuir's isoterm adsorption. The FTIR analysis showed interaction between the EDSD and the surface of the mild steel. The contact angle analysis indicated that the mild steel surface is hydrophobic with the presence of the EDSD. SEM analysis shows a change in the surface of mild steel immersed in a 1 M HCl medium without and with the addition of EDSD.

Keywords: *mild steel, Sambang darah leaves, corrosion inhibitor, Langmuir isotherm, weight loss*

Graphical Abstract



* Corresponding author
Email addresses: emriadi@sci.unand.ac.id

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Introduction

Mild steel is widely used in various fields, such as industry and construction. Despite its good mechanical properties, mild steel corrodes easily in acidic environments [1]. In some attempts to remove rust in industry, strong acid solutions such as sulfuric acid or hydrochloric acid are widely used [2]. Acid solutions are corrosive, so they can damage the surface and accelerate corrosion of mild steel. Therefore, a method is needed to inhibit the corrosion rate [3]. Various methods are used to inhibit corrosion in mild steel, such as coating, cathodic and anodic protection, and the use of corrosion inhibitors. In general, corrosion inhibitors are usually made from organic and inorganic compounds derived from synthetic chemicals, which are expensive, dangerous, and not environmentally friendly [4]. One alternative currently being developed uses inhibitors with plant extracts that are environmentally friendly and economical [5].

Plant extracts are used as corrosion inhibitors because they contain compounds that have heteroatoms such as O, N, or S and aromatic rings that can interact with steel through absorption or acceptance of electron pairs. This interaction results in the formation of coordination bonds or polar covalent bonds, which can prevent corrosion [3]. Several studies have used plant extracts as corrosion inhibitors, such as using leaf extract *Syzygium malaccense* with an inhibition efficiency of 88.11%; leaf extract *Artemisia argyi* with an inhibition efficiency of 96.4%*;* using *Cinnamoum tamala* leaf extract with an inhibition efficiency of 96.76%; and used *locust bean gum* with an inhibition efficiency of 89.8% [5–8].

Excoecaria cochinchinensis L. (EDSD) is a medicinal plant that was traditionally found in eastern Indochina. It has been used to cure several conditions including furuncles, itching, protracted diarrhea, urethrorrhagia, and dysentery [6]. Based on prior phytochemical analyses, the plant has been found to contain various organic components including flavonoids, phenolics, terpenoids, and tannins [7]. This chemical functions as a corrosion inhibitor due to its inclusion of polar functional groups, heteroatoms, and linkages that might hinder corrosion by adhering to steel surfaces [8]. The study seeks to assess the potential of EDSD as a corrosion inhibitor by evaluating its inhibitory efficiency values. Additionally, it aims to provide insights into the management of corrosion in mild steels.

Experimental Section

Materials

The materials used in this research were Sambang Darah leaves from Muara bulian, Batanghari, mild steel (AISI 1020), hydrochloric acid (Smart lab) p.a., deionized water (H_2O), and methanol (CH_3OH) p.a (EMSURE® ACS, ISO, Reag. Ph Eur).

Instrumentation

The equipment used in this research was a rotary evaporator (Heidolph W200), water bath (Innotech BJPX RockFord), analytical balance (Ohaus CP 214), glassware, desiccator, filter paper, iron sandpaper (silicone carbide grit 60). The function group analysis found on the EDSD was performed using the Fourier Transform Infrared (Thermo Scientific Nicolet iS10), to measure the hydrophilicity and hisrofobility properties of the mild steel surface used USB Digital Microscope. (LED50x1000x Wifi Wireless). Morphology of mild steel surfaces analyzed using Surface morphology analysis (HORIBA EMAX x-act).

Procedure

Preparation of mild steel specimens

The surface of the mild steel is smoothed with sandpaper and rinsed with acetone and distilled water. The specimen size is 1 mm thick and measures 3 cm x 2 cm. After that, the oven was heated to 60 °C to dry the mild steel. After drying, the mild steel is weighed expressed as the initial weight (W1) [6].

weight loss measurement

Mild steel was soaked in the corrosive medium HCl 1 M and EDSD with various concentrations (0, 1, 2, 4, and 6 g/L) for 7 hours at temperatures of 30, 40, 50, and 60 °C. After that, the mild steel is cleaned, washed, and dried. After drying, the mild

steel is weighed expressed as the final weight (W2). Calculation of weight loss can be calculated from the difference in weight of the steel before and after being immersed in an acid solution. From the difference in steel weight loss, the corrosion rate and inhibition efficiency can be determined using the following equation [9]:

$$V_{corr} = \frac{\Delta W}{A \times t} \quad (1)$$

Where Vcorr is the corrosion rate (mg cm^{-2} $hour^{-1}$), ΔW is the difference in weight before and after immersion (mg), A is the surface area (cm2), and t is the immersion time (hours). From equation (1) above, the degree of surface coverage (θ) and inhibition efficiency (EI%) can be determined [10,11]:

$$\%EI = \frac{V_{corr(blank)} - V_{corr(ihb)}}{V_{corr(blank)}} \quad (2)$$

$$\theta = \frac{EI}{100} \quad (3)$$

$V_{corr(blank)}$: corrosion rate without inhibitors; $V_{corr(inb)}$ corrosion rate with inhibitors, EI: inhibition efficiency.

FT-IR analysis

Mild steel was soaked for six days in the corrosive medium HCl 1 M with the addition of 6 g/L EDSD. After the mild steel dries, the layer attached to the mild steel is scraped off. After that, FTIR measurements were carried out on the abrasive results of mild steel and pure EDSD [6].

Contact angle analysis

Mild steel was soaked for 7 hours in 1 M HCl solution without and in the presence of 6 g/L EDSD. Then, the surface of the mild steel was dripped with water, and a picture was taken using a USB Digital Microscope camera. Next, the contact angle between the mild steel surface and the water droplets was measured [12].

SEM analysis

Mild steel was soaked for six days in the corrosive medium HCL 1 M without and with EDSD 6 g/L. Then, it is dried, and surface analysis is carried out for mild steel without immersion [13].

Results and Discussions

Weight loss measurement

Figure 1 shows the corrosion rate and inhibition efficiency values for mild steel. It was found that the corrosion rate decreased, and the inhibition efficiency increased with increasing concentration of EDSD. This is caused by the compounds contained in EDSD, namely flavonoids, phenolics, triterpenoids, and tannins, which are adsorbed on the surface of mild steel and form a layer that is resistant to corrosion, thereby reducing the rate of corrosion and increasing the efficiency of inhibition [14,15].

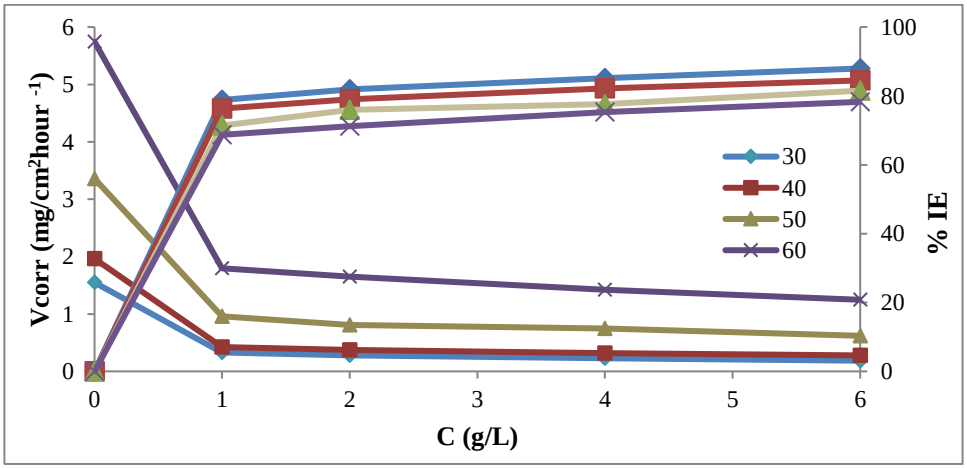


Figure 1. Effect of EDSD concentration on corrosion rate (V_{corr}) and inhibition efficiency (%IE) in 1 M HCl at different temperatures.

The corrosion rate value increases and the efficiency decreases with increasing temperature due to at high temperatures, some EDSD molecules undergo desorption, exposing the steel to the corrosive medium. As a result, the corrosion rate increases, and the inhibition efficiency decreases [15,16]. The highest efficiency value was obtained at an SD concentration of 6 g/L at a temperature of 30°C of 87.972%.

Determination of adsorption isotherms

The adsorption isotherm can explain the adsorption mechanism of corrosion inhibition on the surface of mild steel. Determination of adsorption isotherms using three adsorption methods, namely Langmuir, Freunlich and Temkin isotherms which can be seen in Table 1. It was found that EDSD follows the Langmuir adsorption isotherm because the coefficient of determination is close to 1. The following equation can be used to explain the Langmuir isotherm model [17]:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad (4)$$

Where C is the inhibitor concentration (g/L), θ is the degree of surface coverage, and Kads is the adsorption equilibrium constant.

Figure 2. Shows a straight line C/θ versus C with the EDSD coefficient of determination following the Langmuir adsorption isotherm. It shows that the layer formed is a single layer (monolayer), which means that the molecules are absorbed homogeneously and only in one layer on the surface of the mild steel. In addition, the bond between the compounds in the EDSD and the mild steel is stronger [12].

Tabel 1. Coefficient of determination (R^2) values for various adsorption isotherms.

Temperature (K)	Coefficient of determination (R^2)		
	Langmuir	Freundlich	Temkin
303	0.9993	0.9342	0.9606
313	0.9993	0.9462	0.9934
323	0.9986	0.9557	0.9603
333	0.9985	0.9648	0.9795

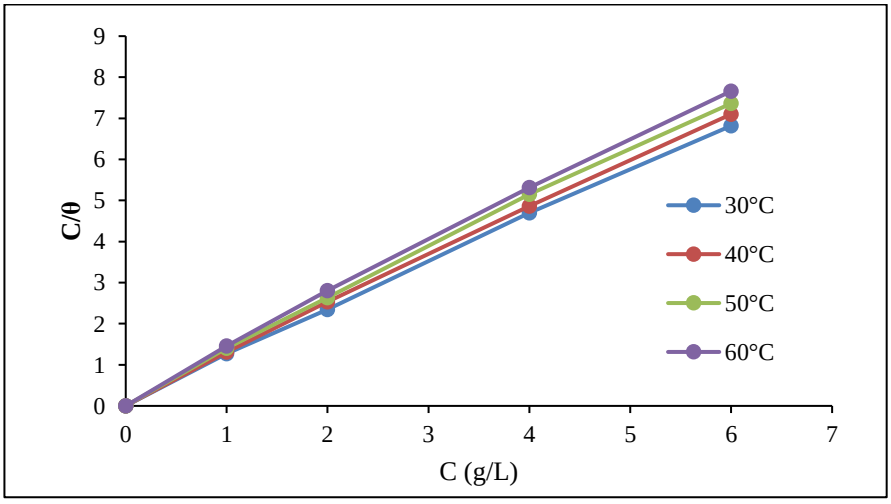


Figure 2. Langmuir isotherm for EDSD adsorption on mild steel in 1 M HCl solution at different temperatures.

The Langmuir adsorption isotherm can also determine the value of the adsorption constant (K_{ads}) from the intercept of the straight-line equation in Figure 2. The K_{ads} value is shown in Table 2. The K_{ads} value increases with increasing temperature due to because the interaction between the EDSD compound and the mild steel surface is getting weaker and causing the compounds in the extract that are bound to Fe^{2+} to be released so that the formation of a protective layer is less and corrosion will occur more quickly [15]. Gibbs free energy can be calculated from the K_{ads} value through the following equation 5.

$$G^{o}_{ads} = RT\ In\ (C_{H_2O} \times K_{ads}) \quad (5)$$

C_{H_2O} (1000 g/L) is in solution and R is the gas constant. The negative value of the Gibbs energy (ΔG^{0}_{ads}) obtained indicates that the adsorption reaction is spontaneous and stable. A ΔG^{0}_{ads} value of less than -20 kJ/mol or more in the positive direction means physical adsorption. If the ΔG^{0}_{ads} value is more than -40 kJ/mol it indicates chemical adsorption. If the ΔG^{0}_{ads} value is between the two, it means it shows a combination of physical and chemical adsorption [10,11]. From the ΔG^{0}_{ads} data values obtained around -23 kJ/mol to -24 kJ/mol and more negative, this shows that adsorption on EDSD is a combination of physical and chemical adsorption and leads to physical adsorption. The ΔG^{o}_{ads} value can be used to determine ΔH^{o}_{ads} and adsorption entropy (ΔS^{o}_{ads}) can be calculated using equation 6 [17, 18]:

$$\Delta S^{o}_{ads} = \frac{\Delta H^{o}_{ads} - \Delta G^{o}_{ads}}{T} \quad (6)$$

The adsorption enthalpy value ΔH^{0}_{ads} obtained is negative, indicating that inhibitor adsorption is an exothermic process, which means that as the temperature increases, the inhibition efficiency value decreases. A positive adsorption entropy value ΔS^{0}_{ads} indicates an increase in solvent entropy. This increase is caused by the adsorption of EDSD molecules which are absorbed more than water (H_2O) [11,13].

Tabel 2. EDSD adsorption isotherm and thermodynamic parameters.

Temperature (K)	K_{ads}	ΔG^{o}_{ads} (kJ/mol)	ΔH^{o}_{ads} (kJ/mol)	ΔS^{o}_{ads} (J/molK)	R^2
303	12.870	-23.835	-17.531	20.704	0.999
313	10.277	-24.037			0.999
323	8.026	-24.140			0.998
333	6.920	-24.477			0.998

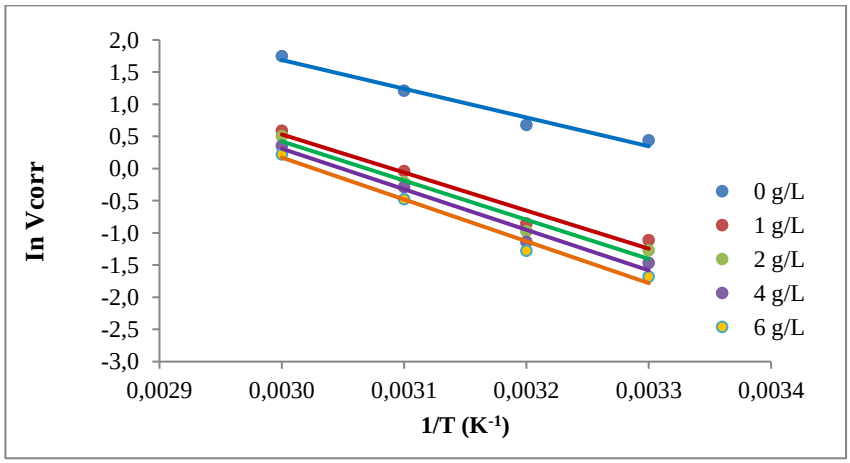


Figure 3. Arrhenius groove 1/T (K^{-1}) vs In V_{corr} without and with the addition of EDSD.

Tabel 3. Activation energy value (E_a), activation enthalpy (ΔH*), and activation entropy (ΔS*).

Inhibitor concentration (g/L)	E_a (kJ/mol)	ΔH*(kJ/mol)	ΔS*(J/molK)
0	37.15	34.53	-128.20
1	49.23	46.61	-101.58
2	50.64	48.02	-98.24
4	52.44	49.82	-93.79
6	54.09	51.47	-86.38

Activation Kinetic Parameters

The plot graph of Arrhenius 1/T (K^{-1}) vs In V_{corr} without and with the addition of EDSD in various concentrations (Figure 3). The straight line with the slope value produced by the graph can be used to calculate the activation energy value, which can be calculated using the Arrhenius equation [19].

$$\text{In } V_{corr} = \text{In } A - \frac{Ea}{RT} \quad (7)$$

Dimana Ea adalah energi aktivasi ($kJmol^{-1}$), A adalah faktor frekuensi (konstanta Arrhenius), R adalah konstanta molar gas (8.314 J $mol^{-1}K^{-1}$), dan T adalah suhu (K)

The E_a value for steel corrosion increases with the addition of EDSD (Table 3). A higher E_a value indicates that more energy is needed for the corrosion reaction to occur. Higher activation values also indicate a physical adsorption mechanism which shows mass and charge transfer, causing a decrease in the corrosion rate [19]. To determine value (ΔH*) and (ΔS*) can be determined using the Arrhenius equation 8 [3,8]:

$$\text{In } \frac{V_{corr}}{T} = \left[\text{In } \left(\frac{R}{Nh}\right) + \left(\frac{\Delta S*}{R}\right)\right] - \frac{\Delta H*}{RT} \quad (8)$$

N is Avogadro's number ($6.023x10^{23}$), h is Planck's constant ($6.63x10^{-34}$), ΔH* is the enthalpy change (J mol^{-1} K^{-1}), and ΔS* entropy change (J mol^{-1} K^{-1}).

Table 3 shows that the (ΔH*) value increases with increasing EDSD concentration. As the value (ΔH*) increases, the corrosion reaction will become more difficult to occur because high energy is required to reach the equilibrium state or activated state [10]. The value (ΔH*) is positive (+), which indicates that the corrosion reaction is endothermic.

The entropy value (ΔS*) obtained increases, indicating that the adsorption of EDSD molecules on the mild steel surface is accompanied by desorption, which increases the entropy of the activation solvent. And the entropy value (ΔS*) is negative (-). It shows that EDSD adsorption slows down the dissolution of mild steel in 1 M HCl solution [10,15].

Functional groups

Secondary metabolite functional groups in EDSD that function as corrosion inhibitors were identified through FTIR analysis. Figure 4 (a) is a pure EDSD spectrum. The spectrum results show that EDSD contains functional groups such as –OH at wave number 3258.35 cm^{-1}, aliphatic C-H group at wave number 2925.57 cm^{-1}, C=O group at wave number 1706.61 cm^{-1}, aromatic C=C at wave number wave number 1514.24 cm^{-1}, C=C alkene group at wave number 1610.04 cm^{-1}, C-O at wave number 1199.64 cm^{-1}, and C-H alkane at wave number 2858.96 cm^{-1} and 1446.95 cm^{-1}.

The results of FT-IR analysis show that the –OH group shifted to a wave number of 3175.36 cm^{-1}, the C-H alkane group shifted to a wave number of 1442.06 cm^{-1}, the C=O group shifted to a wave number of 1689.01 cm^{-1}, the C=C group aromatic shifts to the wave number 1507.93 cm^{-1}, the alkene C=C group shifts to the wave number 1614.52 cm^{-1}, and the C-O group to the wave number 1103.89 cm^{-1} (Figure 4b). This shift in wave number indicates that there is an interaction between EDSD and the surface of the mild steel to form a passive layer on the surface

of the mild steel so that it can reduce the rate of corrosion [11,18].

Contact angle analysis

Contact angle analysis is carried out to determine the degree of wettability between water and the surface of mild steel. Figure 5 (a) shows the surface of mild steel, which was dripped with water after being immersed in 1 M HCl medium and found an angle of 47.183°, which shows hydrophilic properties. The decrease in contact angle is caused by the interaction between the mild steel surface and the 1 M HCl medium, resulting in rust. The surface of rusty steel has pores that can attract water, causing a decrease in the contact angle [20].

Figure 5 (b) shows the surface of mild steel immersed in 1 M HCl medium with the addition of 6 g/L EDSD dripped with water to form a contact angle of 95.997° which shows hydrophobic properties. The increase in contact angle is due to the formation of a protective layer of EDSD which is adsorbed on the surface of the mild steel. This protective layer has hydrophobicity which can inhibit the invasion of aggressive ions that dissolve in water, thereby inhibiting the dissolution process on the mild steel surface [12,20].

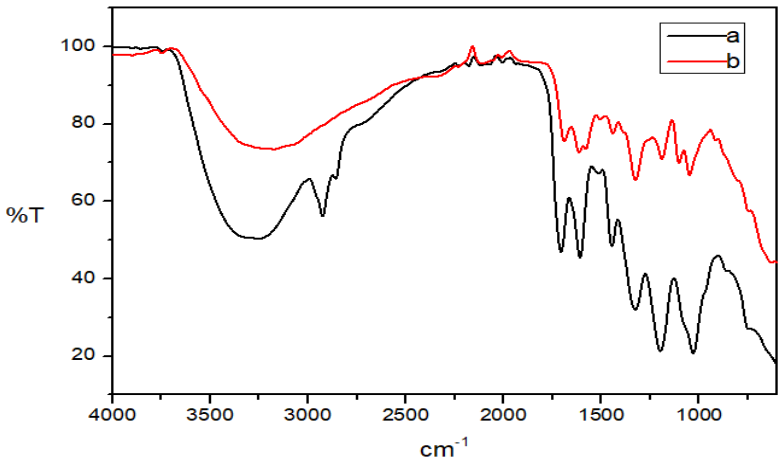


Figure 4. FT-IR spectrum of (a) pure EDSD (b) corrosion production of mild steel in 1 M HCl with the addition of 6 g/L EDSD.

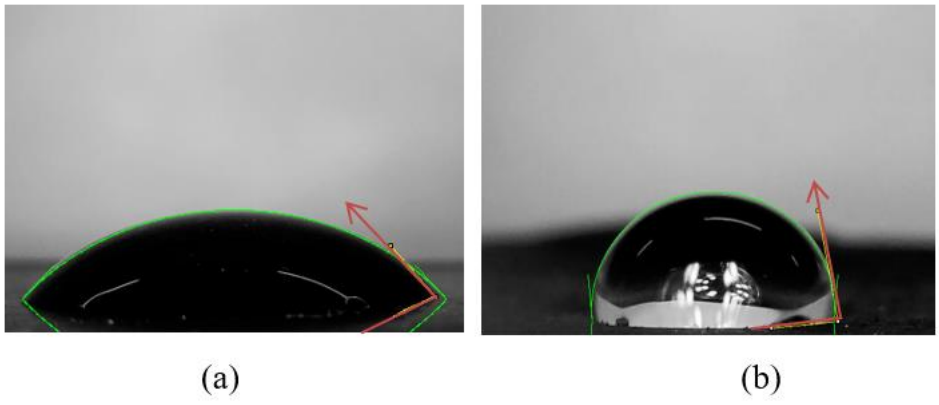
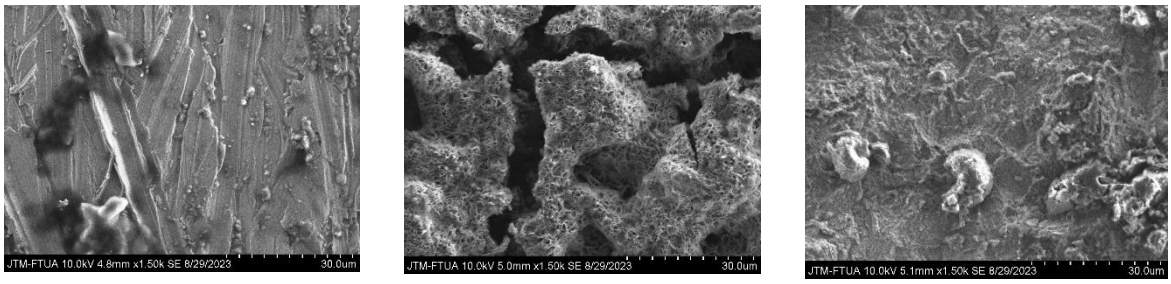


Figure 5. Contact angle of water droplets on the surface of mild steel (a) after being immersed in 1 M HCl solution (b) after being immersed in 1 M HCl + EDSD 6 g/L solution.



(a) (b) (c)

Figure 6. Surface morphology of mild steel (a) without treatment (b) after soaking with 1M HCl (c) after soaking with 1M HCl + EDSD 6 g/L.

Morphological studies

The results of observations of the mild steel surface were carried out with a magnification of 1,500x. The surface morphology of mild steel without treatment, the surface still looks smooth and there are fine lines after sanding and there is no visible damage to the surface because the mild steel has not experienced interaction with 1 M HCl medium (Figure 6a). The surface morphology of mild steel that experiences a corrosion process, which is characterized by the appearance of damage, such as cracks and an uneven surface due to interaction with the 1 M HCl medium, which causes corrosion (Figure 6b).

The surface morphology of the mild steel is flatter, and the addition of EDSD covers the cracks due to the protective layer formed from the adsorption of EDSD on the mild steel surface so that the mild steel surface is better covered (Figure 7c). The protective layer adsorbed on the mild steel surface prevents the corrosion process from occurring [10].

Conclusions

Based on the research that has been carried out, it can be concluded that EDSD can be used as a corrosion inhibitor for mild steel. The highest inhibition efficiency value was obtained at an EDSD concentration of 6 g/L, which was 87.972% at a temperature of 30°C. Adsorption follows the Langmuir adsorption isotherm pattern, which occurs spontaneously. The type of EDSD adsorption is physical and chemical adsorption (mixed adsorption), which is more directed towards physical adsorption.

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