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Date Palm Seed Extract as a Green Corrosion Inhibitor in 0.5 M HCl Medium for Carbon Steel: Electrochemical Measurement and Weight Loss Studies

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In this work, the corrosion inhibiting properties of date palm seed extract (DSE) for carbon steel in 0.5 M hydrochloric acid (HCl) solution has been investigated using several techniques, including weight loss measurement, potentiodynamic polarisation (PDP), and electrochemical impedance spectroscopy (EIS). By adding the DSE concentrations in 0.5M HCI solution from 800 ppm to 2000 ppm, the inhibition efficiency (IE%) was improved with DSE concentration up to 1400 ppm. Highest IE% of 94 in weight loss, 92 in PDP, and 97 in EIS were all attained at an optimum DSE concentration of 1400 ppm. Further increasing the DSE concentration to 2000 ppm, IE% was shown to be decreased. Moreover, the PDP measurement indicated that DSE is a mixed-type inhibitor, affecting both cathodic and anodic partial reactions. From the Gas Chromatography and Mass Spectrometry (GC-MS) analysis, it was revealed that DSE is mainly composed of (wt.%) 36.0 lauric acid, 12.0 caprylic acid, and 10.7 myristic acid. This study revealed that DSE has it could serve as a green corrosion inhibitor for carbon steel in HCl medium.

Keywords: Date palm seed; green corrosion inhibitor; HCl; carbon steel; weight loss measurement; Potentiodynamic Polarisation (PDP) and Electrochemical Impedance Spectroscopy (EIS).

1. INTRODUCTION

Hypernym Carbon steel or hyper-eutectoid steel is one of the exclusive choices of material used extensively in every part of the oil and gas industry, from production and processing to pipeline distribution and transportation facilities [1]. The study of carbon steel corrosion phenomena has gained much attention lately, particularly in acidic media due to the growing manufacturing applications of acid solutions. Acid solutions are widely used in many industries, with a required field of applications such as acid pickling of iron and steel, oil-well acidification, and petrochemical processes. Among the acid media reported in the literature, hydrochloric acid (HCl) is one of the common generally acid-related factors in the pickling of metals, acidification of oil wells, and scales cleaning. The main downfall of using HCl media in all these processes is its inherently poor corrosion resistance and high corrosion rate during the acid treatment process [2]. The presence of corrosive acidic solution at high temperature and pressure environments makes carbon steel-based equipment/pipelines highly vulnerable to a variety of corrosion phenomena. It causes long-term destruction. [3].

Therefore, corrosion inhibitors are one of the standard practical approaches for defending on corrosion and have been used extensively during acid treatment cleaning. The main organic compounds in corrosion inhibitors are mostly heteroatoms, including oxygen, sulphur, and nitrogen, which can be easily absorbed on the steel surface and block active surface sites, decreasing the corrosion rate [4]. Although many industrial inhibitor compounds available up today demonstrated decisive anticorrosive action, however, the largest of it is still very toxic to both human lives and the environment. Thus, the search for environmentally friendly products as corrosion inhibitors has become a new key research area. The desirable vital properties of a renewable and sustainable corrosion inhibitor chemicals must be biodegradable, bio accumulative, and environmentally friendly.

Recently, many researchers have reported a myriad of environmentally friendly corrosion inhibitors developed from plant extracts. Plant extract makes up a substantial class of naturally synthesised corrosion inhibitors aggregates that can be easily extracted from a relatively straightforward procedure at a low in terms of cost. Yet, they are renewable, biodegradable, readily accessible, and harmless to human beings and environments. [5]. An outsized of official papers related to the growth of green corrosion inhibitors in an acidic solution for steel have been published, such as Lannea coromandelica leaf has an IE% of 88 at 250 mg/L. [6], Tabernaemont-ana divaricate an IE% of 95 at 500 mg/L[7], alkaloid extracts from tinctoria and G. ouregou has an IE% of 88 at 250 mg/L[8], the extracts from morus alba, pendula leave has an IE% of 93 at 4000 mg/L[9], bark extracts has an IE% of 79 [10], the extract from quinoline derivatives has an IE% of 97 at 150 mg/L[11]. The extract from Curcumin Longa has an IE% of 89 at 100 ppm in HCl medium[12]. The extract of Kalanchoe Blossfeldina has an IE% of 87 at 300 ppm[13], atenolol drug has an IE% of 94 at 300 mg/L[14], Rollinia occidentalis has an IE% of 86 at 1000 ppm[15], Polysaccharide extract from Plantago has an IE% of 93 at 1000 ppm[16], 700ppm of bauhinia tomentosa leaves has an IE% of 94[17]. Extract of Saraca Ashoka has an IE% of 93 at 100 ppm [18], Red Algae Halopitys Incurvus has a maximum IE% of 87 at 600 mg/L[19]. 600 ppm of Cinnamomum extract has an IE% of 81 [20], Henna Extract has an IE% of 83 at 300 ppm[21]. Extract of Vigna unguiculata, Coat has an IE% of to 84 at 5000 mg/L[22]. Extract of Gongronema latifollium has an IE% achieved to 87 at 1000 mg/L[23]. Thymus satureioides essential oil has an IE% was 89 at 1500 mg/L[24], and the extract of Chamaerops humilis L. fruit extract has a maximum IE% was 79 at 1000 mg/L[25].

Date trees (Phoenix dactylifera) are mainly grown along the Mediterranean coast in North Africa and Arab countries such as Saudi and Iraq. Spain is the only European country where it is built to pass successfully. Date fruits are edible, but the seeds are inedible for humans. Hence, it is most appropriate to utilise the seeds for the production of corrosion inhibitors, which has the potential to replace some organic and inorganic chemical corrosion inhibitors, which are dangerous, harmful, and toxic to humans and the environment. Furthermore, the date palm has been reported to be useful to human health and treatment for sore throats, bronchial catarrh, colds, fever, and gonorrhoea. In the literature review, date palm leaf extract has been used as a corrosion inhibitor in HCl for carbon steel, and an IE% of 80 was successfully demonstrated [26]. Other authors also reported on the anti-corrosion properties of palm fruit extract in NaCl medium, and a maximum IE% of 72 was demonstrated at a concentration level of 2000 ppm for aluminium alloy[27]. In this work, date seeds were used and extracted in an aqueous solution to synthesise a new class of natural plant-based corrosion inhibitors. The IE% of date palm seed extract (DSE) for carbon steel in 0.5M HCl was measured using weight loss measurement, potentiodynamic polarisation (PDP), and electrochemical impedance spectroscopy (EIS) techniques. Furthermore, the corrosion inhibiting activity of DSE was examined in detail from the anodic and cathodic kinetics. Lastly, the chemical composition of extract compounds was analysed and determined using GC-MS analysis.

2. EXPERIMENTAL

2.1. Plant extraction and inhibitor preparation

Date palm seeds used in this study were collected from Baghdad/Iraq. The seeds were firstly washed with water and sun-dried for a few days before further drying in an oven for 20 – 30 min at 40 – 50^{o}C. The dried date seeds were grounded mechanically into powder form. In a typical DSE extraction, 10g of dried date seed powder was mixed with 1L of 0.5M HCl solution. The solution was then heated up to 50 ºC using a hot plate under continuous magnetic stirring for 15h [28]. After 15h, the solution was filtered to eliminate particles of solid suspension before evaporated in a rotary evaporator under reduced pressure. Finally, the extract was diluted with 0.5M HCl to prepare a series of inhibitor stock solutions with different DSE concentrations (800, 900, 1400, and 2000 ppm).

2.2. Preparation of carbon steel

The chemical compounds of carbon steel (wt. %) composed of Fe 99.3, Mn 0.5, P 0.04 C 0.12, S 0.045. The carbon steel was mechanically cut into 2 cm x 2 cm x 2.27 cm dimensions followed by surface abrasion using silicon-carbide paper with #320 - #1200 grades. The clean carbon steel sample was washed with distilled water before stored in a moisture-free dryer box for further use. [29].

2.3. Weight loss measurement

The carbon steel samples were fully immersed in a beaker containing 120 ml of test medium (0, 800, 900, 1400, 2000 ppm). The average reading recorded from three samples were taken from a digital balance, and the nearest reading was rounded up to 3 significant numbers. The carbon steel sample was weighted before immersed in the acid solution. The weighted carbon steel samples were then suspended in different concentrations of the test solution at 25 °C for 72 h. After 72 h, the corrosion test was performed on each sample from the test solution and immersed for 10min in a cleaning solution to remove the corroded products before reweighted again [30]. From the weight loss measurement, the corrosion rates were determined by applying the equation (1)[31]:

$$C_r(mmy^{-1}) = \frac{87600 \times \Delta W}{\rho T A} \quad (1)$$

Where C_r is the corrosion rate, ΔW is the difference between first and final weight for the samples (weight loss), where ρ is the carbon steel density, and A is the surface area of carbon steel (cm^2), while T is the immersion time per hour (h). IE% of DSE was determined by using the equation. (2)[32]:

$$IE\% = \left(\frac{C_{r_{blank}} - C_{r_{inh}}}{C_{r_{blank}}}\right) \times 100 \quad (2)$$

Where $C_{r\ blank}$, C_{inh} is the corrosion rates without/with inhibitor, sequentially. The surface coverage degree (θ) was determined by applying this formula (3)[24]:

$$\theta = \left(\frac{C_{r_{blank}} - C_{r_{inh}}}{C_{r_{blank}}}\right) \quad (3)$$

2.4. Electrochemical measurements

EIS and PDP measurements were conducted to ascertain the corrosive action for carbon steel with/without DSE in 0.5 M HCl using Gamry potentiostat-ZAR Reference-3000, which was connected to the software. The potentiostat was connected to three-electrode cell. Saturated calomel electrode (SCE) was used as a reference electrode. Graphite was used as a counter electrode. Carbon steel was used as the working electrode with an uncovered area of $1cm^2$. The working electrode was placed near to the reference electrode without any contact occurring between the electrodes. PDP curves were created by changing the potential electrode from −250 to +250 mV, opposing the open circuit potential (E_{corr}) at a scan rate of $1mVs^{-1}$. EIS test was also conducted to understand the kinetic and quality of electrochemical procedures in a 0.5 M HCl medium for carbon steel. The carbon steel was immersed in an examination solution for half an hour at open circuit potential (OCP) to achieve a steady-state condition. Electrochemical impedance spectroscopy measurements (EIS) were completed at free open circuit potential (OCP). The range of frequency zone started with 100000 Hz to 0.01 Hz, with 17 points per hertz decade and alternating current (AC) voltage amplitude of ±10 mV. Moreover, software programs controlled the measurements made on the rest potentials at 25^oC automatically. From the PDP measurement, IE% of the inhibitors was measured based on the value of polarisation resistance from the following equation (4)[33]:

$$IE\% = 1 - \left(\frac{I_{corr}}{I^{o}_{corr}}\right) \times 100\% \quad (4)$$

Inhibition efficiency percentage (IE%) of EIS data were calculated by comparing the charge transfer resistance values with/without DSE using equation (5):

$$IE\% = \left(\frac{R_{ct} - R_{ct}^{\circ}}{R_{ct}}\right) \times 100\% \qquad (5)$$

2.5. Gas chromatography-mass spectrometry (GC-MS)

The chemical composition of DSE was analyzed using a gas chromatograph-mass spectrometer (GC-MS). The GC-MS was conducted using an Agilent 7890A gas chromatograph (GC) coupled with a mass spectrometer system (MS) of an Agilent 5975C inert Mass Selective Detector (MSD) among triple-axis detector, model DB-5MS UI, length(L) 30m while the film thickness 0.25µm and Diameter 0.25mm. The MSD Chemstation was employed to obtain the peaks in the GC raw. The operating conditions of GC-MS are recorded in Table 1.

Table 1. GC-MS instrument control parameters used to examine the DSE.

GC		MS	
Carrier gas	Helium	Transfer line temperature	250^{o}C
Inlet temperature	280^{o}C	Source temperature	220^{o}C
Mode split-less	Flow	Solvent delay	6.00 min
Injection volume	1 µL	Low mass	45.0
Injection type	Standard	High mass	600.0
Average velocity	36.445 cm/sec		
Pressure	7.6522 psi		
Run time	5.03 min		

3. RESULTS AND DISCUSSION

3.1. Weight loss measurements

To investigate the effect of DSE concentrations on IE% and Cr for carbon steel in 0.5M HCl, the weight loss of carbon steel sample was held after 72h immersion time. Figure 1 illustrates the effect of IE% and Cr as a function of DSE concentrations (ppm). From Figure 1, DSE exhibited excellent corrosion inhibiting properties as all DSE extract concentrations were capable of inhibiting the corrosion activity of carbon steel in 0.5M HCl. Table 2 presents the weight loss of carbon steel in 0.5M HCl. From Table 2, both ΔW and Cr parameters vary inversely. IE% improved considerably with increasing DSE concentration up to an optimum DSE concentration of 1400 ppm. The Cr parameter was found to be declined significantly from 2601 mmy^{-1} to 152 mmy^{-1} after 1400 ppm, and the corresponding optimum IE% was determined as 94. When the DSE concentration increased further to 2000 ppm, IE% decreased slightly to 93. This could be because the mild dissolution of excess molecular hydrogen gas in HCl medium and the permeation of molecular hydrogen through the steel membrane, causing a small loss of collected molecular hydrogen. Therefore, IE% value was found to be reduced at high inhibitor concentration. This trend is in great harmony with other similar works summarized in the literature [34].

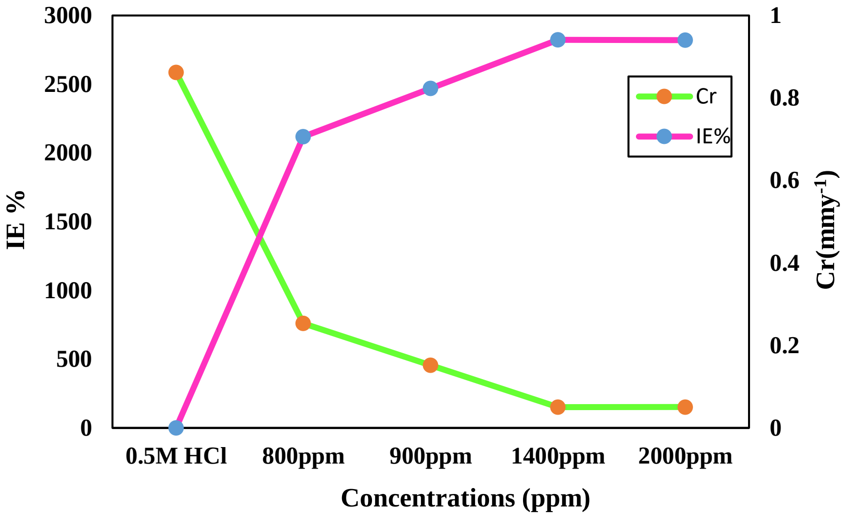


Figure 1. Corrosion rate C_r and inhibition efficiency IE% in 0.5M HCl for carbon steel with respect to DSE concentrations from 0.5M HCl – 2000 ppm.

Table 2. Weight loss of carbon steel in 0.5M HCl at 0.5M HCl-2000 ppm DSE after 72h immersion time.

Concentrations (ppm)	ΔW(mg)	C_r (mmy^{-1})	θ	IE%
0.5M HCl	171	2601	-	-
800ppm	50	760	0.71	71
900ppm	30	456	0.82	82
1400ppm	10	152	0.94	94
2000ppm	11	167	0.93	93

3.2. Electrochemical test

3.2.1. Potentiodynamic-polarization (PDP)

Potentiodynamic studies were performed to investigate the anodic and cathodic reactions progress ($2H^+ + 2e \longrightarrow H_2$), ($Fe \longrightarrow Fe^{2+} + 2e$) [35]. Figure 2 indicates the PDP curves in 0.5M HCl for carbon steel. Figure 2 shows that the anodic metal dissolution and cathodic-hydrogen development evolution were inhibited in the presence of DSE. The parameters obtained from the polarisation experiments, including corrosion current density (I_{corr}), corrosion potential (E_{corr}), cathodic Tafel slope (β_c), and anodic Tafel slope (β_a) are presented in Table 3. From Table 3, the I_{corr} parameter decreased with an increase of inhibition concentration up to 1400 ppm. On the other hand, IE% also improved as

DSE's concentration level increased from 800 ppm to 1400 ppm, reaching a maximum IE% of 92. On a further increment extraction to 2000 ppm, IE % started to decrease slightly.

The slight shift of cathodic Tafel slope β_c can be explained due to the influence of inhibitor compounds on the kinetics of hydrogen-evolution in the presence of the DSE inhibitor. Whereas, the changes of anodic Tafel slope β_a values could be attributed to the adsorption of DSE molecules onto the carbon steel surface and blocking the active surface sites. Furthermore, the E_{corr} of carbon steel was observed to shift towards less negative values. when the movement in E_{corr} value is more than 85 mV, the inhibitor can be categorized as an anodic or cathodic inhibitor [36]. From our results, the inhibitor did not shift the E_{corr} values significantly, and thus, the DSE can be concluded as a mixed type inhibitor. These outcomes are in accord with those observed in earlier studies [37].

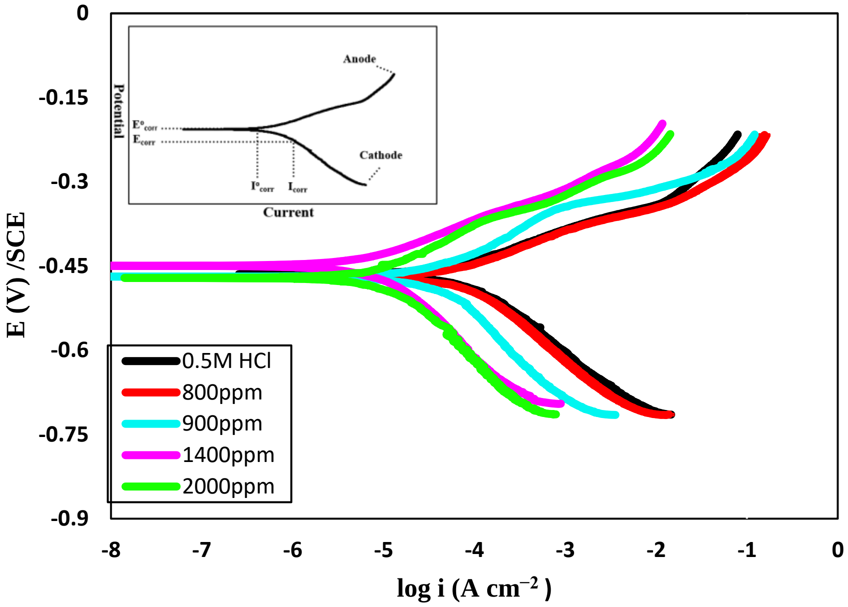


Figure 2. Polarisation curve for carbon steel in 0.5 M HCl with several concentrations of DSE.

Table 3. Polarisation parameters for carbon steel in 0.5M HCl with several concentrations of DSE.

concentrations	I_{corr} (μAcm^{-2})	E_{corr} (mV/SCE)	B_a (mV/dec)	B_c (mV/dec)	IE%
0.5M HCl	84	-464	80	126	-
800ppm	30	-470	60	119	64
900ppm	20	-469	65	123	76
1400ppm	7	-450	67	138	92
2000ppm	8	-471	134	73	91

3.2.2. Electrochemical-impedance spectroscopy (EIS)

EIS was performed to identify the dynamic characteristics and electrochemical procedures of carbon steel in HCl medium and provide a clear elucidation on how DSE inhibitor compounds modified them. Figure 3(a) and (b) show the Nyquist plot and Bode plot sequentially with/without DSE. The width of the capacitive loop becomes more extensive when the concentration level of DSE is increased up to 1400 ppm. The Nyquist plot is connected to R_s, which is the solution resistance, and the low-frequency catch with the real axis ascribed to the R_{ct}, which is the charge transfer resistance [38]. The Nyquist plot consists of one capacitive curve in the high-frequency zone, which linked to the one time constant in the Bode plot [39]. Figure 3(c) illustrates the electrochemical equivalent circuit that applied to analyze the EIS result. It contains R_s among the moving electrode and a reference electrode. In contrast, R_{ct} is the charge among metal and outside, and CPE is the constant phase factor.

IE% values were calculated using equation 5, and the values are displayed in Table 4. The result showed that IE% improved significantly with developing concentrations of DSE up to 1400 ppm with a maximum IE% of 97. Then, IE% decreased considerably at 2000 ppm.

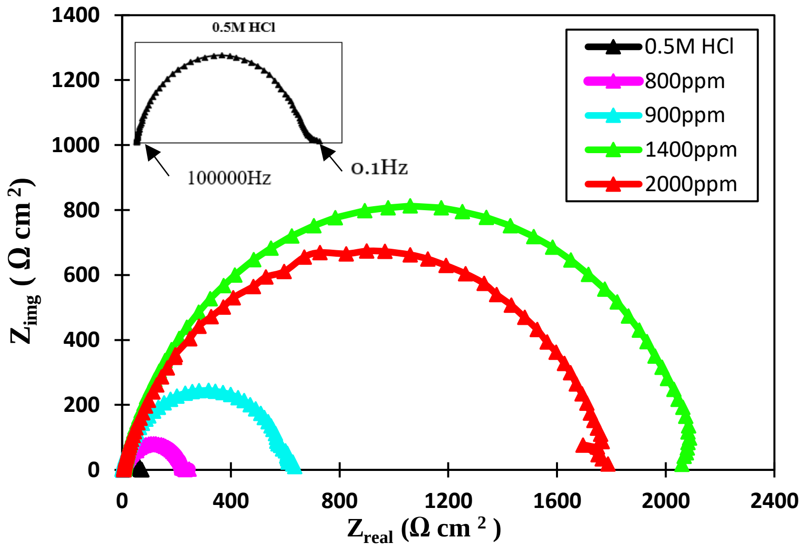
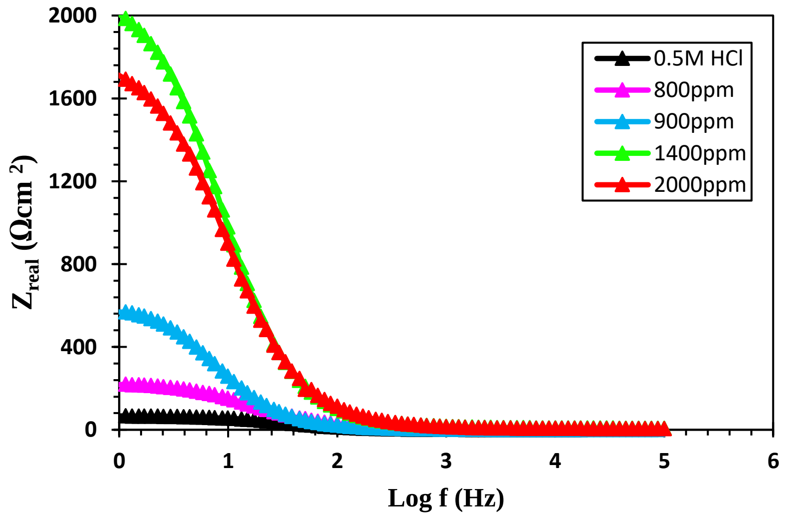


Figure 3(a). Electrochemical Impedance Nyquist plot for carbon steel with different concentrations of DSE.



Figurer 3(b). Electrochemical Impedance Bode plot for carbon steel in o.5 M HCl with several concentrations of DSE.

The undesirable bonding interactions can explain this among the adsorbed molecule on the steel surface and the freely moving molecules that occupied at the interface of inhibitors solution. The freely mobile inhibitor molecules interrupt the ionic bonding between the absorbed molecules and the steel surface. The freely molecules pull away from the adsorbed molecules from the steel surface with their steric hindrance and repulsive forces. This subsequently caused little inhibitor molecules available on the metal surface, protecting it from corrosion activity and leads to a decrease in IE%. Table 5 compares some of the parameters and IE% values achieved from three techniques (weight loss, PDP, and EIS). One can notice that IE% values are very much consistent with each other's and share a similar optimum DSE concentration level at 1400 ppm. Table 6 also compares the IE% values of DSE and other plant extracts used as corrosion inhibitors for different steel in HCl medium at place temperature.

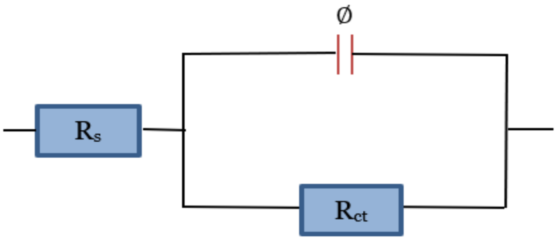


Figure 3(c). Equivalent circuit parameters applied to fitting experimental data.

Table 4. Electrochemical Impedance parameters for carbon steel in 0.5M HCl with different concentrations of DSE.

concentrations	R_{ct} (Ω cm^2)	R_s (Ω cm^2)	IE%
0.5M HCl	66	622	-
800ppm	228	1	71
900ppm	607	626	89
1400ppm	2098	7	97
2000ppm	1771	5	96

Table 5. A comparison of the IE% results from three methods (weight loss, PDP, EIS).

	Weight loss		PDP		EIS	
concentrations	C_r (mmy^{-1})	IE%	I_{corr} (μAcm^{-2})	IE%	R_{ct} (Ωcm^2)	IE%
0.5M HCl	2601	-	84	-	66	-
800ppm	760	71	30	64	228	71
900ppm	456	83	20	76	607	89
1400ppm	152	94	7	92	2098	97
2000ppm	167	93	8	91	1771	96

Table 6. Comparing of corrosion IE% values of DSE with different plants extracts for various steel in HCl medium.

				Weight loss	PDP	EIS	
Plant	Steel type	Concentration	Immersion time for weight loss test	IE%	IE%	IE%	Reference
Date palm seeds extract	Carbon steel	1400ppm	72h	94	92	97	This work
Tabernaemont-ana divaricate extract	Carbon steel	500ppm	2h	95	91	89	[7]
Curcumin longa oil extract	Al	100ppm	3h	89	89	88	[12]
rollinia occidentalis extract	Carbon steel	1000ppm	3h	72	86	80	[15]
Polysaccharide from plantago	Carbon steel	1000ppm	6h	89	93	94	[16]
Bauhinia tomentosa leaves extract	Mild steel	700ppm	2h	93	94	93	[17]
Red algae extract Halopitys incurvus extract	Carbon steel	600ppm	6h	82	87	88	[19]
Cinnamomum extract	Carbon steel	600ppm	3h	80	81	73	[20]
Henna extract	Carbon steel	300ppm	3h	82	83	80	[21]
Vigna unguiculata, Coat extract	Mild steel	5000ppm	24h	87	84	83	[22]
Gongronema latifollium extract	Mild steel	1000ppm	24h	91	87	92	[23]
Thymus satureioides essential oil extract	Mild steel	1500ppm	6h	90	89	87	[24]
Chamaerops humilis L. fruit extract	Mild steel	1000ppm	6h	88	79	78	[25]
Date Palm leaf extract	Mild steel	2000ppm	24h	91	80	88	[26]

3.3. Gas chromatography-mass spectrometry (GC-MS)

Figure 4(a) presents the fatty acid compositions of DSE obtained from GC/MS. Gas chromatography (GC) is the method widely applied to identify the quantification of non-polar group products. Table 7 presents ten fatty acid peaks and their respective weight (%). Lauric acid is indicated as peaks 1, 5, 7, and 9. The analysis showed that the total fatty acid composition in DSE was more than 80.0%. Figure 4(b) depicts the fatty acid concentrations in terms of percentage. Lauric acid is the primary fatty acid compound with the highest percentage (wt. %) of 36.0, followed by caprylic acid 12.0, myristic acid10.7, oleic acid 9.3, phthalic acid 8.7% and lastly palmitic acid 3.0. This is in excellent harmony with the previous work reported in the literature [40]. The chemical structure of DSE was identified through a stable spectral library structure, as shown in Figure 4(c). From Figure 4(c), one can be observed that a variety of covalent bonds involving the transfer of electrons from the inhibitor to the metal surface are formed. The intensity of the chemisorption bond is governed by the electron density of the functional-group and polarizability group linked to the donor atom. Electron-donating substituents like (– CH) improves the availability of donor electrons at the steel surface, forming an enhanced protection layer. Molecular structure with more than nine carbon atoms such as lauric acid, myristic acid, oleic acid, and palmitic acid has a better corrosion IE% due to the tremendous effort in these rings, which can be relieved from the development of a robust chemisorption bond on the steel surface [41].

Table 7. The fatty acid compositions of DSE

Peak	Identified compounds	Formula	Weight (%)
1	Lauric acid	$C_{12}H_{24}O_2$	17.8
2	Myristic acid	$C_{14}H_{28}O_2$	10.7
3	Oleic acid	$C_{18}H_{34}O_2$	9.3
4	Phthalic acid	$C_8H_6O_4$	8.7
5	Lauric acid	$C_{12}H_{24}O_2$	8.0
6	Caprylic acid	$C_8H_{16}O_2$	7.0
7	Lauric acid	$C_{12}H_{24}O_2$	6.5
8	Caprylic acid	$C_8H_{16}O_2$	4.8
9	Lauric acid	$C_{12}H_{24}O_2$	3.7
10	Palmitic acid	$C_{16}H_{32}O_2$	3.0

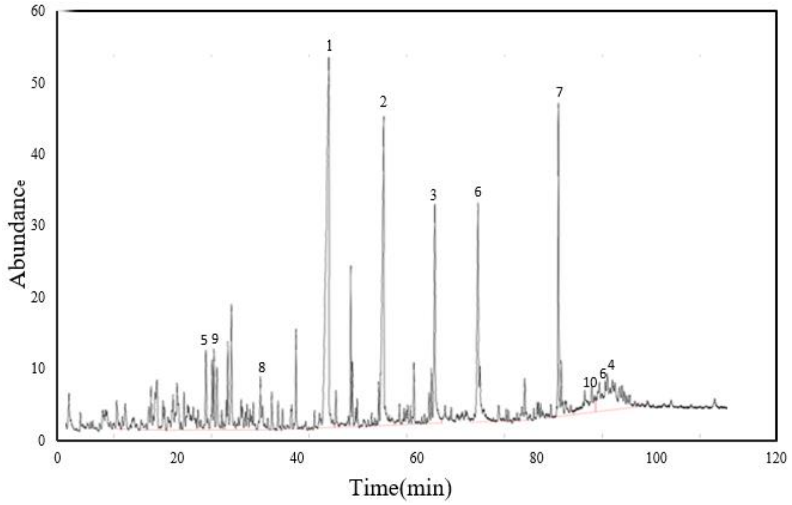


Figure 4(a). Diagram of DSE by GC-MS.

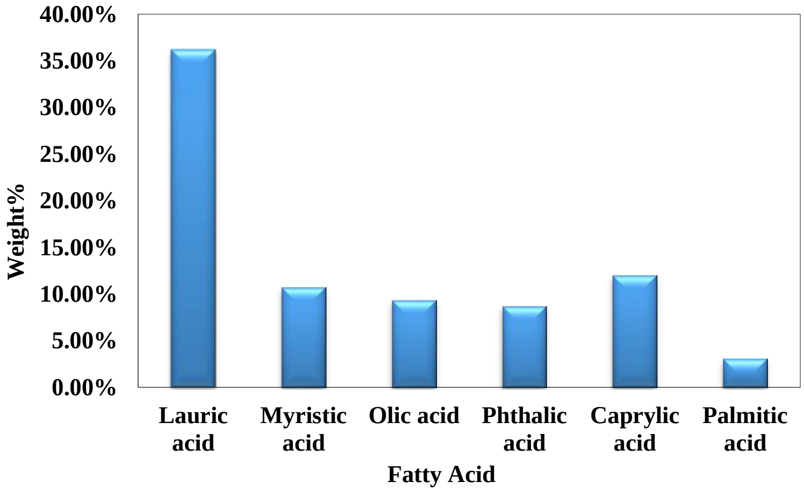


Figure 4(b). Fatty Acid type as a function of concentrations in DSE.

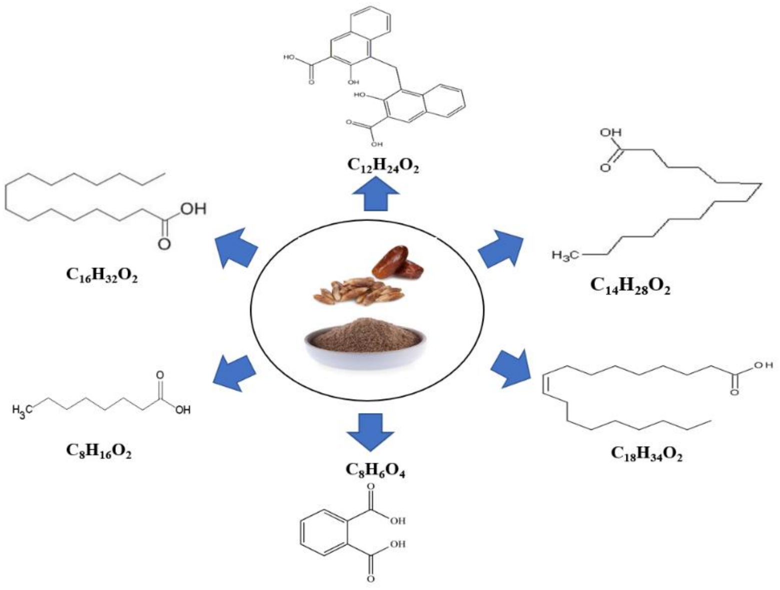


Figure 4(c). Fatty acid structures of DSE

4. CONCLUSION

In this work, DSE extract excellent corrosion inhibiting properties in 0.5M HCl for carbon steel. From the results, the highest IE% was obtained at an optimum DSE concentration of 1400 ppm with weight loss, PDP, and EIS measurements of 94, 92, and 97, respectively. Besides, DSE inhibitors were found to be capable of inhibiting the carbon steel corrosion activity by blocking the active position and reducing the corrosion rates. Apart from that, the DSE inhibitor is a mixed type inhibitor because the difference of E_{corr} values before and after adding the inhibitor was less than 85 mV. From the GC-MS results, DSE mainly composed of (wt. %) lauric acid 36.0, caprylic acid 12.0, myristic acid 10.7, oleic acid 9.3, phthalic acid 8.7 and palmitic acid 3.0.

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