

Date Seed Extract as Green Corrosion Inhibitor for Steel in Hydrochloric Medium

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Abstract

The date seed extract is considered an effective and environmentally friendly corrosion inhibitor. In this study, the ability to inhibit corrosion of steel in an acidic medium was tested by measuring weight loss, potentiodynamic polarisation, scanning electron microscope techniques, and Fourier Transform infrared spectroscopy. Depending on weight loss, it was observed that inhibition efficiency increased with inhibitor concentration, while the corrosion rate decreased. The maximum inhibition efficiency was 93% for 0.6g/L in 0.5M HCI. The results from the polarization curves showed that the extract behaved as a mixed-type corrosioninhibitor, and the maximum inhibition efficiency was 99% for 0.4 and 0.6g/L in 0.5M HCI. The molecule inhibitor adsorption isotherm plots showed that the adsorption of the extract follows the Langmuir isotherm. Scanning electron microscope techniques emphasized the prevent corrosion of the metal surface due to the adsorption of inhibitor molecules on its surface.

Paper type: Research paper

Keywords: Date seed, corrosion inhibiter, steel, adsorption isotherm, Potentiodynamic, polarization.

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Introduction

Corrosion has negative implications in three main areas: economics, safety, and environmental degradation. Metallic corrosion, which appears to be harmless, has a significant impact on many areas of the economy. Because the great corrosion costs substantially impact the national economy, corrosion personnel must implement corrosion control techniques to avoid losses. One of the most significant consequences of corrosion is the loss of safety. While industrial workers should prioritise safety, accidents do happen. According to the NACE International Bulletin (Hansson, 2011). Annual corrosion losses in the United States are projected to be in the billions of dollars. Corrosion is not just costly but also dangerous to human life. Corrosion of hulls of iron on ships, for example, the resulting loss of those hulls, poses a threat to the crew. Chemical industries that handle corrosive chemicals that release cyclohexene are more prone to accidents. Corrosion can also harm the environment. The environmental consequences of oil or gas pipelines or tank corrosion are serious, such as water and air pollution, which can lead to the extinction of aquatic life. Accidents caused by corrosion can theoretically harm irreplaceable wildlife (Fontana, 1963; Koch, 2001; Gerhardus, 2016).

Corrosion is the gradual deterioration of a material caused by chemical and electro-chemical reactions with its environment and is a result of the inherent tendency of metals to revert to their more stable compounds, usually oxides. Most metals are found in nature in the form of various chemical compounds called ores (Speight, 2014).

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The manufacturing process to convert these ores to metals is endothermic meaning that it involves energy input. Nevertheless, the corrosion reaction is exothermic, meaning that added energy will be released, and the metal is returned to its oxide state (Van Delinder, 1984; Affonso, 2007). Metallic corrosion can be divided into the first type dry chemical corrosion is defined as the reaction of metals with dry air or oxygen. In the presence of electrolytic solutions, wet or electrochemical corrosion is common. One of the solutions to this problem is a corrosion inhibitor it is a substance that when introduced to a corrosive media, reduces the metal's corrosion rate, inhibition can be caused by a delay in either the anodic or cathode processes or both. Corrosion inhibitors should have the following properties:

- (1)Strong adsorption onto the surface of the metal.
- (2)Reducing the degree of reactant diffusion to the surface of the metal.
- (3)Lowering the metal surface's electrical resistance.

Corrosion inhibitors are more commonly used, the inhibition could be due to chemical and/or physical adsorption of these molecules on the metal surface, which results in the creation of a protective barrier film (Al-Mashhadani, 2020). Every year, the number of date seeds produced increases. Furthermore, pitted date exports are on the rise, and large quantities of date seeds are produced. For manufacturing businesses, this biomass is an issue. Date seeds are commonly wasted or used as animal feed ingredients. However, due to its valuable chemical makeup, this co-product can be employed for a variety of additional purposes. Date seeds contain 5-13% oil. Saturated and unsaturated fatty acids are found in date seed oil, with lauric and oleic being the most prevalent. Significant levels of tocopherols, tocotrienols, phytosterols and phenolic compounds are also present. Date seeds contain several phytochemicals, which can be employed in a variety of applications, including food product formulations, cosmetics, and pharmaceuticals (Mrabet, 2018). The date seed has long been one of the most significant fruit crops in the southern Mediterranean region, where dates constitute a major source of income and a staple diet for many locals. Furthermore, this crop is not only a source of economic gain but also vital to stabilizing populations and establishing or preserving centres of life (Christoph, 2000; Habib, 2009; Rahman, 2007).

Corrosion inhibitors are one of the most widely used corrosion control strategies, corrosion inhibitor safety and environmental concerns have been always a global concern. Even though many synthetic chemicals have anticorrosive properties, they are expensive and highly harmful to humans and the environment. The natural product has been widely used as green corrosion inhibitors of metals in aggressive media because they are more environmentally friendly, less expensive, and simply available. The present research purpose is to investigate some natural compounds known as Date seeds to be used as corrosion inhibitors for steel in Acid solution (HCl). Thus, the research involves the examination of the inhibiting effect of Date seed extracts toward the corrosion of steel and the Table 1 Chemical composition of E316L-16 stainless steel.

determination of the type of adsorption isotherm to determine the percentage inhibition efficiency for Date seed extracts.

1 Materials and Methods

1.1 Chemical composition of steel

The experiments were performed using stainless steel (type E316L-16) in acid
solutions. The chemical composition of this type of stainless steel is given in
Table 1.

1.2 Chemical 1.2.1 Inhibitor preparation

Elements	Chemical composition (weight %)
Fe	63.27
Cr	18.7
Ni	12.6
Mn	1.75
Si	0.52
Р	0.04
S	0.03
Cu	0.75
Mo	2.30
С	0.04

Date Seed in this study was washed with distilled water several times and left

to dry at room temperature, then a 20g portion was taken and ground into a powder (2µm). The powder Date Seed Extract (DSE) was soaked in concentrated methanol for four hours. The mixture was filtered using filter paper to get the cake. The prepared powder DSE was distributed into different portions and dissolved in distilled water in 250ml volumetric flasks to prepare the solution with different concentrations namely 0.1, 0.2, 0.4, and 0.6g/L.

1.2.2 Solutions Preparation

A corrosive solution of 0.5M HCl was prepared by diluting analytical grade 37% HCl with bi-distilled water in a 250ml volumetric flask as the final volume. The inhibitor was dissolved in the acid solution at the desired concentration and the solution in the absence

of the inhibitor was taken as blank. The test solutions were freshly prepared before each experiment by adding DSE directly to the corrosive solution at different concentrations (0.1, 0.2, 0.4, and 0.6 gL).

1.3 Electrochemical measurements

The electrochemical measurements were carried out using a Gamry instrument potentiostat. All potentials given in this study refer to this reference electrode (Ag/AgCl or Calomel). The working electrode was immersed in the test solution for 30 minutes to establish steady-state open circuit potential (E_{ocp}). After measuring the E_{ocp} , the electrochemical measurements were performed. The Tafel polarisation curve was obtained in the potential range from -0.2 to 0.2V with 2mV\s scan rates. The corrosion cell had three electrodes as follows in **Figure 1**.



Fig.1 Explain the circuit of potentiostat a)Gamry instrument potentiostat reference 600, b) Work electrode is steel electrode 1.2x3 cm, c) reference electrode which is Ag/AgCl or saturated Calmel, d)Auxiliary electrode which is platinum.

1.3.1 Work Electrode

A steel electrode of 1.2x3cm was plumped from one side to an electric wire connection. Before measurement, the surface of the working electrode was mechanically abraded using a different grade of emery papers, which ended with the 1000 grade. The specimen was cleaned by washing it with bi-distilled water, thoroughly degreasing it with acetone, washing it once more with bi-distilled water and finally dry in it with filter paper.

1.3.2 Reference Electrode

A saturated calomel electrode was used during potentiodynamic polarisation measurements. The electrical was checked to contact with electrolyte to be free from any bubble.

1.3.3 Auxiliary Electrode

Platinum was selected to provide a current to the electric cell as a counter electrode.

1.3.4 Weight loss measurements

Steel sheets of 36mm were washed with distilled water, degreased with acetone, dried and kept. After weighing them accurately with a highly sensitive digital balance, the specimens were immersed in a solution containing 0.5M HCl with and without various concentrations of DSE. After two hours of exposure at 25°C, the specimens were taken out, dried and weighed accurately again.

1.3.5 Surface morphology studies

The surface morphology of the steel specimens was examined after exposure to 0.5M HCl with and without the selected inhibitor. A Scios 2 HiVac SEM was used for this investigation.

2 Results and Discussion

2.1 Chemical structure for DSE by FT-IR

The DSE was analysed via FT-IR spectroscopy for the disintegration of the functional group in a molecule of organic and inorganic compounds. **Figure 2** shows the FT-IR absorption spectrum peak for the extracts in thisspectrum. The peak appears to be at $3400cm^{-1}$ corresponding to the stretching mode of hydroxyl (OH) for alcohol or phenol and secondary amine at $3742cm^{-1}$. Fig. 2 presents FT-IR absorption spectrum peak at $1630cm^{-1}$ corresponding to the stretching mode of the stretching mode

These peaks indicate that the extracted solution contains this functional group, which acts as the centre of the electron, helps the inhibitor adsorb on the metal surface, and acts as an anti-corrosion reagent. However, the heteroatoms, such as nitrogen and oxygen, numerous bonds are the main adsorption centres on the metal surface due to their greater basicity and electron density. Inhibition is due to the chemical or physical adsorption of these molecules on the metal surface, which creation a protective barrier film. This explains why plant extracts incessantly contain different organic molecules with various functional groups (Amuda, 2011), these functional groups are rated as the strongest corrosion resistance groups. It has been inferred that those heteroatoms, aromatic rings, and electrons enhance the adsorption of corrosion inhibitors. Moreover, the polar functional groups make the inhibitor more soluble in the corrosive medium and tend to act as extension sites for the corrosion inhibitor molecule (Du Toit, 2007; Ahamad, 2010; Gopiraman,

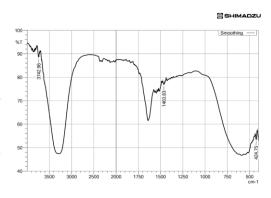


Fig. 2 IR spectra of date seed extracts.

2012; Muralisankar, 2017; Zhang, 2016). The spectrum confirms that the DSE contain numerous compounds, such as secondary amine, phenolic and carboxylic acids. These compounds contain heteroatoms (N, O). These atoms have electronic density and can adsorb on the metal surface via coordinated bonds.

2.2 Weighing Loss Method

The corrosion protection performance of the inhibitor was determined by *WL* measurement, all specimen values are reported in **Table 2** The values of *WL*, corrosion rate (*R in g*/cm.min) and corrosion inhibition efficiency *IE* (%) are given in Table 2 and **Figures 3**, **4** and **5**, *R* and *IE* of each concentration and the average weight lost Δw were *W*1 and *W*2 are the average weight of specimens before and after exposure. calculated using the following equations (Zaher, 2020).

$$\Delta w = W_1 - W_2 \tag{1}$$

$$\theta = \frac{R_{un} - R_{in}}{R_{un}}$$
$$IE\% = \frac{\frac{R_{un} - R_{in}}{R_{un}} \times 100$$

where ΔW is the average WL (mg), S is the surface area of specimens (cm²), T is the immersion time (min), θ is the surface coverage, and R_{un} , R_{in} are corrosion rates in the absence and presence of the inhibitor, respectively. According to Table 2, the *IE* increased with increasing inhibitor concentrations, while the corrosionrate decreased. This behaviour can be attributed to the increase of surface coverage due to the adsorption of the inhibitor on the steel surface. The maximum inhibition efficiency was 93% for 0.6g/L in 0.5M HCl acid. The effectiveness of DSE as a corrosion inhibitor is due to its chemical composition of antioxidants, saturated and unsaturated fatty acids and phenolic compounds. The results confirmed the excellent performance of DSE on the corrosion inhibition of mild steel in 0.5M HCl solution as a

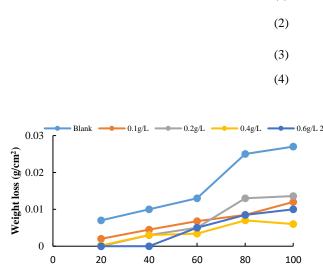


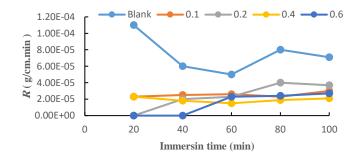
Fig. 3 The relationship between weight loss and immersion time for steel in 0.5M HCl in the absence and presence of different concentrations of DSE at 25°C.

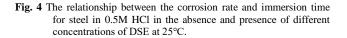
Immersin time (min)

corrosive medium. The optimum concentration required to achieve an efficiency of 93.3% was found at 0.6 g/L (Zaher, 2020).

Time (min)	Inhibitor Concentration (g/L)	Weight loss (g)	Corrosion Rate(g/cm.min)	<i>θ</i> (-)	IE (%)
20	0	0.008	0.00011	-	-
	0.1	0.002	0.000028	0.75	75
	0.2	0.002	0.000030	0.72	72.72
	0.4	0.002	0.000028	0.75	75
	0.6	0.002	0.000028	0.75	75
40	0	0.010	0.000139	-	-
	0.1	0.004	0.000056	0.60	60
	0.2	0.004	0.000061	0.56	56.36
	0.4	0.003	0.000042	0.70	70
	0.6	0.004	0.000028	0.81	80
60	0	0.013	0.000181	-	-
	0.1	0.006	0.000083	0.53	53.84
	0.2	0.005	0.000076	0.58	58.04
	0.4	0.004	0.000056	0.69	69.23
	0.6	0.005	0.000023	0.87	87.17
80	0	0.024	0.000333	-	-
	0.1	0.008	0.000111	0.66	66.66
	0.2	0.010	0.000152	0.54	54.54
	0.4	0.007	0.000097	0.70	70.83
	0.6	0.008	0.000028	0.91	91.66
100	0	0.027	0.000375	-	-
	0.1	0.015	0.000139	0.62	62.96
	0.2	0.011	0.000167	0.55	55.55
	0.4	0.007	0.000097	0.74	74.83
	0.6	0.009	0.000025	0.93	93.33

Table 2 Corrosion parameters of mild steel in 0.5M HCl solution with and without different concentrations of DSE.





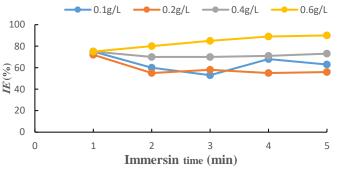


Fig. 5 The relationship between the inhibition efficiency and immersion time for steel in 0.5M HCl in the different concentrations of DSE at 25°C.

2.3 Potentiodynamic polarization measurements

The potentiodynamic polarisation curves of the steel in 0.5M HCl with and without the addition of different concentrations of DSE are shown in **Figure 6**. Corrosion current density of the blank I_{corr_o} , Corrosion current density of inhibitor $I_{corr_{inh}}$, corrosion potential E_{corr} , anodic Tafel slope β_{α} and cathodic Tafel slope β_c represented in these experiments' results in **Table 3**. Fig.6 shows that the anodic (solution of steel) side is slightly more shifted than the cathodic (hydrogen reaction) side. These observances

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indicate that the inhibitor displays cathodic and anodic inhibition effects incessantlyDSE is a mixed type of inhibitor, which means that it, will be effective at both anodic and cathodic corrosion inhibition for steel. Table 3 shows that an increase in DSE concentration led to a decrease in the corrosion current anodic Tafel slope and the cathodic Tafel slope of the DSE inhibitor will be shifted to a more positive and negative area on the potential curve. This suggests that the inhibitor molecules are adsorbed on the metal surface by blocking the active sites on the metal surface and stripping the molecule inhibitor, also retarding the corrosion reaction. The inhibition efficiency IE values increased concentrations of DSE on the surface of steel up to 99.9% at 0.4 and 0.6g/L. IE is calculated according to the following equation (Cox, 1996).

 $\% IE = \frac{I_{corr_{\circ}} - I_{corr_{inh}}}{I_{corr_{\circ}}} \times 100$

Inhibitors	Concentration (g/L)	-E _{corr} (mVSCE)	β, a (v/dec)	β , c (v/dec)	I _{corr} (Acm ⁻²)	IE (%)
DSE	0.1	842.4	81.86	309.6 x 10 ⁻³	9.353	32.85
	0.2	730.7	43.7x10 ⁻³	8.63x10 ⁻¹²	1.189	91.46
	0.4	655.1	46x10 ⁻³	$18.82 \mathrm{x10^{-3}}$	$1 x 10^{-4}$	99.9
	0.6	556.9	64.34x10 ⁻³	55.53 x10 ⁻³	$1 x 10^{-4}$	99.9
Blank	0	13.37	2.752	1.615	13.93	-

Table 3 Potentiodynamic polarisation parameters for the corrosion of steel in 0.5M HCl solution with and withoutdifferent concentrations of DSE at

2.4 Adsorption isotherm

The mechanism of corrosion inhibition can be explained using Langmuir adsorption isotherm.

$$\frac{c_{inh}}{\theta} = \frac{1}{\kappa_{ads}} + C_{inh} \tag{6}$$

Where C_{inh} the inhibitor concentration, K_{ads} is the adsorptive equilibrium constant and θ is the surface coverage for different concentrations of DSE in 0.5M HCl using the flowing equation.

$$\theta = \frac{R_{un\,inh} - R_{inh}}{R_{un\,inh}} \tag{7}$$

A straight line as described in Figure 7 is obtained from plotting $\frac{C_{inh}}{\theta}$ against C_{inh} , $R_2 > 0.99$ indicates that the adsorption of DSE obeys the Langmuir isotherm. In addition, K_{ads} can be determined by the intercept of the straight line. The adsorptive equilibrium constant K_{ads} is related to the adsorptive standard free energy ΔG°_{ads} , which can be calculated by the following equation: $\Delta G^{\circ} = -RT \ln (1 \times 10^6 K_{ads})$ (8)

Where R and T are the standard gas constant and thermodynamic temperature respectively, and 1×10^{6} is the concentration of water molecules expressed in mgL⁻¹. The K_{ads} and ΔG°_{ads} values are listed in **Table 4** Generally if the value of ΔG°_{ads} is less than -20kJmo l^{-1} , the adsorption is regarded as physisorption, in which the adsorption is due to the electrostatic interaction between the inhibitor molecules and iron atoms. However, if the absolute value of ΔG°_{ads} is 40 kJ mol-1 or higher, the adsorption can be regarded as chemisorption in which a covalent bond is formed due to the transfer and/or share of electrons from inhibitors to metal surfaces. The calculated value of ΔG°_{ads} is approximately -50.69kJ mol⁻¹, indicating that the adsorption of DSE on the steel surface involves

Fig. 6 Polarisation curves for steel in 0.5M HCl containing different concentrations of DSE at 25°C.

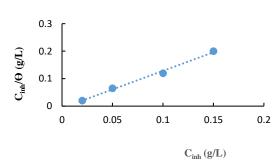
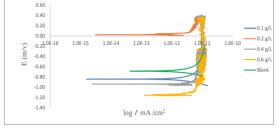


Fig. 7 Langmuir adsorption curve of DSE on the steel surface in 0.5M HCl at 25°C after 100 minutes.



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both chemical and physical adsorption (Zoob, 1985).

2.5 Scanning Electron Spectroscopy Measurements

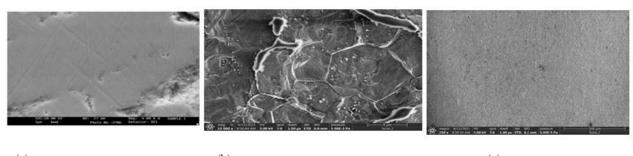
The SEM images obtained in **Figure 8** determined several molecule components of the extract with the surface metal before and after immersion for 120 minutes in 0.5M HCl in the presence and absence of DSE. The shown in (Fig. 8.a) displays the smooth surface of the metal. This represents the absence of any corrosion inhibitor or products formed on the metal surface. When the specimen was immersed in 0.5M HCl (Fig. 8.b) it developed a coarse and harsh surface

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Table 4 Equilibriu	m constant and ac	dsorptive standard free
energy at	25°С.	
Temperature (K)	$K_{\rm ads}$ (L mg ⁻¹)	$\Delta G^{\circ}ads$ (kI mol ⁻¹)

Temperature (K)	$K_{\rm ads}~({\rm L.mg}^{-1})$	$\Delta G^{\circ}ads$ (kJ mol ⁻¹)
298	0.769	-50.69

covered with corrosion products and full of cavities and pits. (Fig. 8.c) presence at a high concentration of DSE 0.6g/L, the surface coverage by extract components increases, which causes the insoluble complex to form on the surface of the metal. This indicates that the extracted molecule prevents the dissolution of iron by forming an adsorbed layer (Fauvet, 2012).



(a) (b) (c) Fig. 8 SEM images of steel (a) alone, (b) after exposure to 0.5M HCl and (c) after exposure to 0.5M HClcontaining 0.6 g/L of DSE.

Conclusions

The inhibition efficiency of DSE on the corrosion of steel in 0.5M HCl solution increased with inhibitor concentration, while the corrosion rate decreased. The maximum inhibition efficiency was 93% for 0.6g/L in 0.5M HCl. Potentiodynamic polarisation observed that the extracted display anodic and cathodic inhibition effects, meaning that the DSE is a mixed type of inhibitor. The maximum inhibition efficiency was 99% for 0.4 and 0.6g/L in 0.5M HCl. FT-IR indicate that the extract contains different functional groups, such as primary amine, phenol, carboxylic acid and others that act as the centre of electron density. That helps the inhibitor more soluble in the corrosive medium. Adsorption of the inhibitor molecules of DSE on the steel surface obeys Langmuir adsorption isotherm. SEM techniques assured that the DSE molecule adsorbed on the surface of the steel and prevented corrosion.

Nomenclature

C C_{inh} DSE ΔG_{adso} ΔW	=concentration =inhibitor concentration =Date Seed Extract =adsorptive standard free energy =average weight lost	[g/L] [g/L] [-] [kJ/mol] [mg]
ΔWL	=average weight lost Method	[g/cm ²]
E_{corr}	=corrosion potential	[mVSCE]
FT-IR	=Fourier-Transform Infrared Spectroscopy	[1/cm]
HCL	=hydrochloric acid	[M]
Icort	=corrosion current density	$[A/cm^2]$
I _{corr} °	=corrosion current density without inhibitor	$[A/cm^2]$
IE	=corrosion inhibition efficiency	[%]
Kads	=adsorptive equilibrium constant	[L/mg]
β_a	=anodic Tafel slope	[v/dec]
β_c	=cathodic Tafel slope	[v/dec]
R	=corrosion rate	[g/cm.min]
R	=universal gas constant	[J/mol.K]

Rin	=corrosion rate in the presence of the inhibitor	[g/cm min]
Run	=corrosion rate in the absence of the inhibitor	[g/cm min]
S	=surface area	[cm ²]
SEM	=Scanning Electron Spectroscopy	[-]
Т	=thermodynamic temperature	[K]
t	=immersion time	[min]
θ	=surface coverage	[L]
θ	=surface coverage for different concentrations of DSE	[L]
W1	=weight of specimens before exposure to 0.5M HCl	[mg]
w2	=weight of specimens after exposure to 0.5M HCl with inhibitor	[mg]

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