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Characterization of Dwarf Palm Leaf Extract (DPLE) (*Chamaerops Humilis L.* Extract) as an Eco-Friendly Corrosion Inhibitor for Carbon *XC70 Steel* in a 3.5% NaCl Solution

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ABSTRACT

In the present work dwarf palm leaf extract (DPLE) has been prepared to be used as a corrosion inhibitor on carbon steel in a saline medium. The inhibitory efficiency of DPLE on the corrosion rate of carbon XC70 steel (CS) in a 3.5% NaCl solution has been studied by means of weight loss measurements, potentiodynamic polarization curves, electrochemical impedance spectroscopy, SEM and AFM microscopies. The results showed that the corrosion inhibition rate of carbon XC70 steel in the NaCl solution increases with the concentration of DPLE, and reaches up to 90% at $2.0{\cdot}10^{-4}$ $g.L^{-1}$ as the optimum concentration of DPLE. The inhibiting performance against corrosion was attributed to the formation a DPLE barrier that reduces the contact area between the carbon XC70 steel and the corrosive solution. The EIS analysis revealed that the presence of DPLE was found to decrease the double layer capacitance, with an increasing charge transfer resistance. The morphological analysis showed that upon adding DPLE in saline solution, the surface morphology of the metal becomes smoother due to the formation a protective layer adsorbed on the metal surface. This study showed that dwarf palm leaf extract acts as an efficient and eco-friendly inhibitor on carbon steel in saline medium.

1. INTRODUCTION

Corrosion represents a fundamental natural process wherein materials undergo deterioration due to complex physicochemical interactions with their environment [1,2]. This phenomenon induces alterations in their properties and a functional decline, particularly observable in steel materials exposed to aggressive environments commonly used in industries such as oil and gas production [1]. Here, infrastructures including pipelines, face constant susceptibility to degradation, primarily from aqueous solutions rich in salts accompanying hydrocarbon extraction, often laden with CO_2 and H_2S. These compounds substitute corrosive conditions for steel equipment [3].

Recent advancements in corrosion inhibition approaches have driven research toward exploring eco-friendly alternatives to synthetic inhibitors. Green non-toxic inhibitors have gained significant interest in industrial corrosion protection. Accordingly, exploring the effect of plant extract-based inhibitors on carbon steel corrosion presents a promising solution in this research field [4].

The exponentially increasing use of plant extracts as corrosion inhibitors is due to their potential efficacy, cost-effectiveness, and environmental sustainability [5, 6]. These plant extracts include phenolic acids, flavonoids, as well as minerals and vitamins. These compounds can adhere to the metal surface via functional groups, forming a protective layer against aggressive media for the metal [7]. HPLC-MS characterizations indicated the presence of 21 and 23 phenols in leaf and pollen extracts, respectively. Furthermore, leaf extracts were found to contain polyphenols identified by comprehensive two-dimensional liquid chromatography (LC × LC), which could be an effective source for corrosion inhibition [8]. Studies conducted on various plant extracts, such as Raphia palm extract in citric acid [9], Spondias Mombin Leaves Extract on the Corrosion of Aluminium Alloy (AA2024) and Mild Steel in 0.5M NaCl [10], Aloe vera extract in seawater [11], Persea schiedeana Ness extract for brass surfaces [3], and fig leaf extract for steel in hydrochloric acid showcase promising inhibitory effects and mechanisms [12]. The exploration of botanical sources like plants leaves as green corrosion inhibitors further underscores the potential of plant-based solutions in industrial applications [13]. This shift towards sustainable and effective corrosion inhibition using plant extracts sets the stage for a comprehensive investigation into their mechanisms and optimization for diverse steel corrosion scenarios [14,15].

We found that most new research focuses on studying the behavior of natural corrosion inhibitors in a saline medium, so we decided to study and evaluate the effectiveness of natural corrosion inhibitors in salt media. In this work investigation dwarf palm leafs extract have been chosen as corrosion inhibitors because of their non-toxic nature and have an eco-friendly compounds . To investigate their inhibiting action on the corrosion of carbon *steel XC70* in 3.5% NaCl solution using weight loss, electrochemical techniques (impedance spectroscopy (EIS) and potentiodynamic polarization). SEM and AFM techniques were used for the morphological analysis of carbon *steel XC70* samples.

2. EXPERIMENTAL

2.1 Materials

The materials that were used for this work included dwarf palm leaf extract (Chamaerops humilis L.) DPLE, carbon steel XC70 (CS), and a solution of NaCl.

2.1.1. Extraction of dwarf palm leaves

The Chamaerops humilis L. or dwarf palm leaves (DPL) used were collected in April 2023 in the wilaya of Biskra, Algeria. The leaves were washed and dried in the shade for fifteen days and stored in paper bags, away from light and moisture until their use. The dried leaves were then ground using a blender until a powder is obtained. The extraction process was carried out using a Soxhlet extractor, where 10 g of dried powder was added to the 50 mL/50mL of distilled water /ethanol for 4 hours at 50°C. After Soxhlet extraction, the dwarf palm leaf extract DPLE obtained was concentrated into a dark green paste using a rotary evaporator at 75°C. The preparation steps described above are shown in Figure 1.

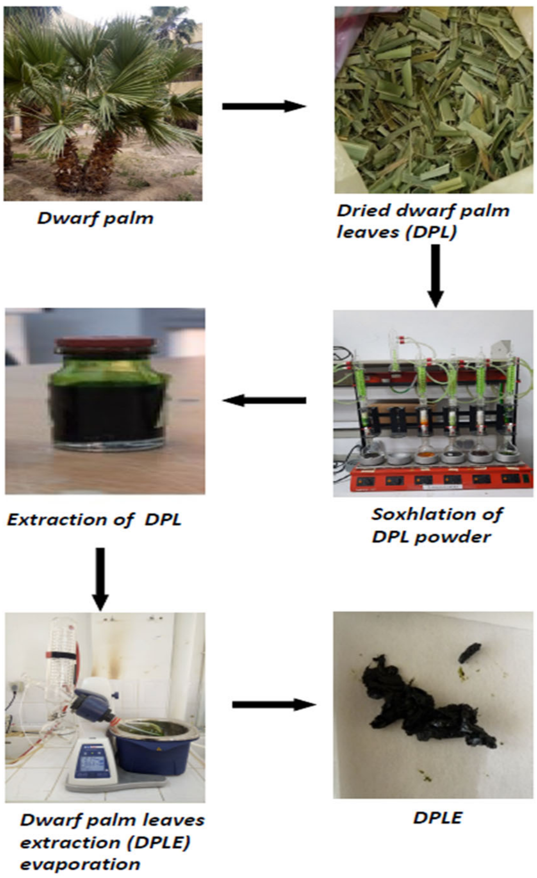

Figure 1. Steps of the extraction of the corrosion inhibitor from *dwarf palm leaves(DPL).*

2.1.2. Characterization of plant extract

The plant extract (DPLE) was screened for alkaloids, tannins, flavonoids and polyphenols, using a Qualitative Phytochemical analysis. The phytochemical analysis was carried out based on protocols adopted by several researches in this field [8], [16-19]. DPLE has also been analyzed using GC-MS-QP2020 NX (SHIMADZU) to determine its composition. A chromatograph model GC-2030 coupled with a mass spectrometer. Extraction was performed on a capillary column DB-5MS (30 m × 0.25 mmID, 0.25 µm). The carrier gas was Helium with a flow rate of 1.5 mL/min. The column temperature was programmed to rise from 40 to 200°C at the rate of 4°C/min. The temperature of both injector and detector was set at 250°C to ensure efficient vaporization and detection of the analytes. A sample volume of 1 µL was injected using a split mode with a split ratio of 30:1, and the injector temperature was set to 220°C.

The functional groups present in the prepared extract were characterized using FTIR spectrometer model Agilent Cary 630, equipped with an FTIR/ATR-DRIFT device.

2.1.3. Preparation of the electrolyte

The electrolyte solution was prepared as a corrosive medium (3.5% NaCl). The dwarf palm leaf extract (DPLE) was dissolved in the 3.5% NaCl solution to prepare three different concentrations as follows: 10^{-4}g.L^{-1}, 2.10^{-4}g.L^{-1}, and 3.10^{-4}g.L^{-1}.

2.1.4. Preparation of steel samples

The carbon XC70 steel (CS) had a chemical composition of 0.125% C, 0.27 % Si, 1.68% Mn, 0.051% Cr, 0.04% Ni, 0.05% S, 0.021% Mo, 0.045% Cu, 0.003%Ti, 0.038% Nb, 0.038% P, 0.012% Al, and balance Fe. Carbon steel 1.0 cm × 1.0 cm × 1.0 cm cubes were used for the weight loss measurements, the morphology analysis and electrochemical tests. The steel samples were prepared by polishing the material surface with abrasive paper using progressively decreasing grain sizes ranging from 200 to 3000. Then, a 1µm diamond paste was applied for rinsing with distilled water and acetone to remove the dust and grease. The carbon steel surface was finally dried under flowing air.

2.2. Effect of the inhibitor on the corrosion behavior on carbon XC70 steel in 3.5% NaCl solution

2.2.1. Weight loss measurements

The gravimetric analysis required the immersion of the CS samples in 200 mL of 3.5% NaCl solution, for 24 hours in ambient environment, without and with variable concentrations of DPLE. The experiment was repeated twice. The weight loss (*W)* of the samples was determined by calculating the difference between the initial weight W_0 and the final weight W_f after immersion in the solution.

$$W = W_0 - Wf \quad (1)$$

The surface coverage (θ), inhibition efficiency (*EI in* %) and corrosion rate (*Cr*) were calculated using the following equations:

$$\theta = \frac{W_0 - Wf}{W_0} \quad (2)$$

$$EI\% = \frac{Cra - Cre}{Cra} . 100 \quad (3)$$

$$Cr = \frac{87{,}6.W}{SDT} \quad (4)$$

Where, *W* is the weight loss of carbon steel (mg), S the size of the piece (cm^2) (surface of the cube exposed to corrosion), *t* is the exposure time (h), and D the density of steel ($g.cm^{-3}$).

2.2.2 Electrochemical measurements

In order to investigate the inhibitory effectiveness of DPLE, electrochemical analyses were conducted.The electrochemical methodologies employed included potentiodynamic polarization (PDP), linear polarization resistance (PR), and electrochemical impedance spectroscopy (EIS), utilizing a PGP201 Potentiostat. Software (Voltamaster 4) was used for evaluating the experimental data. A three-electrode electrochemical cell configuration was utilized, comprising a reference electrode (RE) of Ag/AgCl3 saturated in KCl, a platinum auxiliary electrode (AE), and the XC70 steel sample as the working electrode (WE) with a surface area of $1cm^2$. The WE was immersed in a 3.5% NaCl solution, both with and without DPLE, for 1hour to establish a stable open circuit condition.

From the potentiodynamic polarization method, the inhibition efficiency (IEp) was calculated using the following equation (5):

$$IE_{p(\%)} = \frac{i_{corr} - i_{corr\,inh}}{i_{corr}} x100 \qquad (5)$$

Where, i_{corr} and $i_{corrinh}$ are the corrosion current densities of the uninhibited and inhibited metal steel samples (CS) respectively.

The inhibition efficiency (IE_R) obtained from linear polarization resistance study was calculated using the following equation:

$$IE_{R(\%)} = \frac{Rp_{corr} - Rp_{corrinh}}{Rp_{corr}} x100 \qquad (6)$$

Where, Rp_{corr} and $Rp_{corrinh}$ are polarization resistance of the uninhibited and inhibited metal steel specimens of carbon XC70 steel respectively.

2.2.3. Morphological analysis SEM and AFM of metal steel

Scanning electron microscopy (SEM) was carried out to analyze the composite morphology. SEM analyse was performed using a Thermo Scientific Quattro Environmental Scanning Electron Microscope (SEM, Thermo Fischer Scientific, USA). The accelerating voltage ranged from 200 V to 30 kV. The surface roughness of XC70 steel exposed to corrosive media without and with different concentrations of DPLE has been observed using an atomic force microscope (AFM) manufactured by Bruker.

3. RESULTS AND DISCUSSION

3.1. Characterization of dwarf palm leafs extract (DPLE)

3.1.1. Phytochemical Screenings of DPLE

The phytochemical screenings of dwarf palm leaf Extract (DPLE) are summarized in table 1. Most of these components (phenols, tanins alkanoids ...etc) are heterogeneous organic compounds containing nitrogen, oxygen, sulfur and/or aromatic structures [20]. The presence of flavonoids is characterized by their three aromatic ring structure linked by single and double bonds, and varying with the functional groups attached to the basic structure in the molecular structure. These components may play an important role as effective inhibitors of metal corrosion [21].

Table 1. Phytochemical Screenings of dwarf palm leaves Extract (DPLE)

Phytochemicals	Qualitative analysis
Alkaloids	+
Flavonoids	+
Polyphenols	+
Tannins	+
Saponins	-

Notes: +, present; –, absent

3.1.2. Gas chromatography-mass spectrometry analysis of DPLE

Gas chromatography-mass spectrometry (GC-MS) spectra of DPLE are shown in the figure 2 and table 2 indicates that DPLE contains ethyl esters, Phenol, fatty acids such as palmitic, stearic, oleic, linoleic [9,22]. These components are known to be adhesive at the carbon steel surface [23, 24]. It can be stated that the majority of its constituents exhibit favorable anti-corrosion properties.

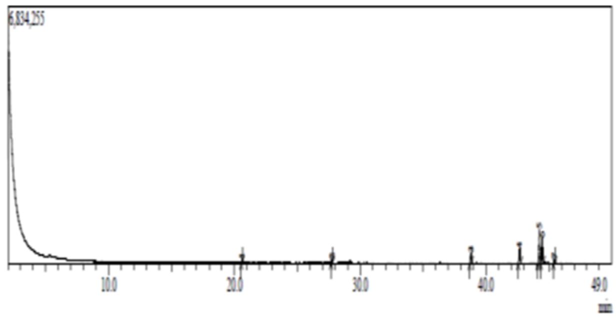

Figure 2. GC-MS spectra of DPLE

Table 2. Phytochemical composition of the DPLE by GC-MS.

Peak	R. Time	Component	Type	Area (%)
1	20.595	Carvacrol	-	0.97
2	27.737	Stearic acid	fatty acid	1.83
3	38.790	Palmitic acid	fatty acid	21.73
4	42.652	4-isopropyl-1,7,11-trimethyl-2,7,11-cyclododecatriene	Cyclic molecule	16.21
5	44.227	linoleic acid	fatty acid	28.49
6	44.453	Oleic acid	fatty acid	26.72
7	45.416	2-methyl-5-(1-methylethyl)phenol	Phenol	4.05

3.1.3. Fourier transform infrared spectroscopy

The FTIR spectrum for DPLE is illustrated in Figure 3. The presence of a broad absorption band at 3200 cm^{-1} is attributed to O-H and N-H. It is related to the presence of the hydroxyl and amide groups in the DPLE [25]. The presence of two absorption bands between 2924 cm^{-1} and 2853 cm^{-1} show the C–H stretching. The vibration band confirmed the C=O bond at 1700

cm^{-1}. At 1600 cm^{-1}, C=C stretching vibrations of aromatic ring were recorded. An asymmetric N–O stretching vibration at 1500 cm^{-1} indicates the presence of a nitro compound.

The stretching vibration of the C–C bond was found at 1410 cm^{-1}. The strong band at 1030 cm^{-1} is attributed to the C–O stretching vibration of carbohydrates. Other absorption bands located at 710 cm^{-1} are assigned to N–H stretching vibrations [12]. Therefore, it can be inferred that these functional groups have the potential to be adsorbed and utilized to obstruct active metal centers. These functional groups have the ability to hinder the metal corrosion by forming a protective surface film that isolates the metal from the corrosive environment. Polar functional groups and structural double bonds can serve as adsorption sites during interactions with metal inhibitors [26].

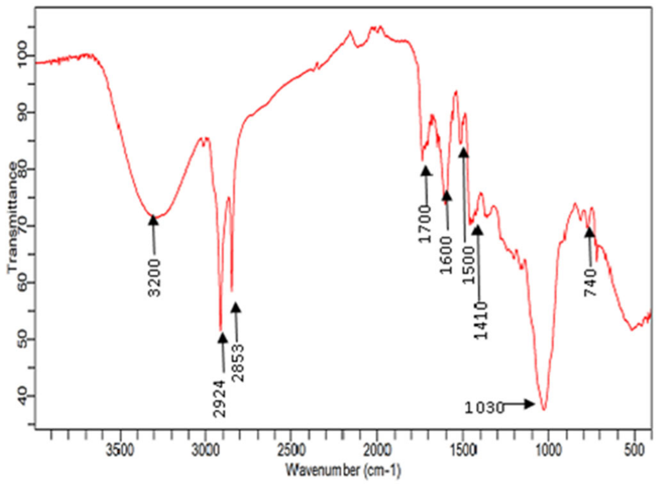

Figure 3. FT-IR Spectra of *Dwarf palm leaf* Extract

3.2. Characterization of metal steel (XC70)

3.2.1. Weight loss measurements

Corrosion rate and inhibition efficiency of XC70 steel in 3.5% NaCl solution with and without dwarf palm leaf extract (DPLE), expressed by weight loss values after 24 hours of immersion, are listed in Table 3.

The results in Table 3 present the weight variation indication the corrosion rate of CS immersed in 3.5% NaCl solution with varying the DPLE content (C_{DPLE}), as well as the immersion time. The corrosion rate is reduced in the presence of DPLE. Furthermore, the higher the extract concentration; the lower the corrosion rate. This supposes that the extract molecules have been adsorbed on the metal creating a protective layer on the metal surface [24,27].

The highest value of inhibition efficiency was 92.92%, after 24 hours of immersion, at a concentration of 2.10^{-4} $g.L^{-1}$ of DPL extract. The results in Table 3 showed that increasing the concentration of DPLE above 2.10^{-4} $g.L^{-1}$ led to an increase in the corrosion rate and a decrease in the inhibition efficiency. Which suggests the concentration of DPLE of 2.10^{-4} $g.L^{-1}$ as the ideal concentration for the higher corrosion inhibition effectiveness [3].

Table 3. Gravimetric parameters of XC70 at 298 K in 3.5% NaCl solution with and without DPL extract.

C_{DPLE} (g/l)	24 hours	
	Cr (mm/y)	EI (%)
Blank	1.694	-
10^{-4}	0.223	86.78
2.10^{-4}	0.057	92.92
3.10^{-4}	0.4404	74

3.2.2. Electrochemical studies

The polarization curves obtained for CS immerged in a 3.5% NaCl solution, without and with various concentrations of dwarf palm leaf extract (C_{DPLE}), are illustrated in Figure 4. The corrosion potential (E_{corr}), corrosion current densities (icorr), Tafel's cathodic and anodic slopes (βc and βa), and the inhibition efficiency (EI%) obtained are given in Table 4.

Figure 4 shows the Tafel polarization plot regarding CS in 3.5% NaCl solution with various contents of DPLE at 298 K. The addition of Chamaerops Humilis L. extract at different concentrations induces slight modifications in the corrosion potential values. These values exhibit non-uniform variations, and both cathodic and anodic current densities decrease with increasing concentration, except at the concentration of 3.10^{-4} $g.L^{-1}$, where an increase in current density is observed. These findings indicate that the inhibitor exerts both cathodic and anodic effects, suggesting that the compound acts as a mixed inhibitor, due to the shift in Ecorr value less than 85 mV [12,17]. Also, the upward trend in the slopes of both anodic and cathodic Tafel lines (βc) and (βa) with the increasing concentration of DPLE suggests a decrease in the rate of hydrogen ion reduction due to the presence of the inhibitor. This results in a higher energy barrier for proton discharge. This trend indicates that the DPLE compound influences the kinetics of hydrogen reduction and metal dissolution. The compound is supposed initially to adsorb onto the metal surface, blocking certain sites without affecting the anodic reaction mechanism. The addition of DPL extract resulted in a significant decrease in corrosion current density (icorr) and increasing Rp values [28-31], which is indicative of an efficient inhibition of carbon XC70 steel corrosion in 3.5% NaCl solution. This introduces the potential of DPL extract as a promising corrosion inhibitor for challenging environments.

The inhibitory capacity of DPLE increases with increasing the concentration, reaching its highest value (90.66%) at 2.10^{-4} g L^{-1}. These results support the significant inhibitory action of DPLE regarding CS corrosion in saline solution (3.5%NaCl).

Table 4. Polarization parameters of CS in 3.5%NaCl solution in the absence and presence of DPLE.

C DPLE (g/l)	Rct Ω. cm^2	Q_{dl} µF.cm^{-2}	EI_R (%)
Blank	127.1	175.2	-
10-4	327.4	153.6	61.17
2.10-4	421.0	75.59	69.80
3.10-4	349.4	81.05	63.63

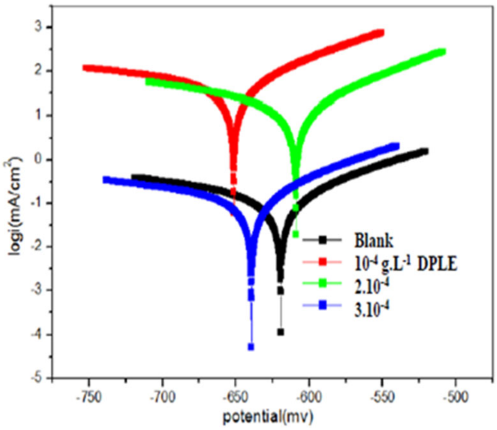

Figure 4. Tafel polarisation plot of CS in 3.5%NaCl solution without and with different DPLE concentrations.

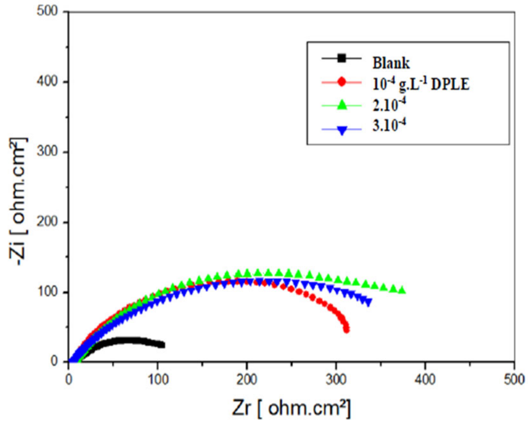

Figure 5. Nyquist curves of CS in 3.5% NaCl solution without and with different DPLE concentrations.

Table 5. EIS parameters of CS in 3.5% NaCl solution with and without DPLE.

C_{DPLE} (g/l)	Blank	10^{-4}	$2.\ 10^{-4}$	$3.\ 10^{-4}$
Ecorr (mV/Ag/AgCl)	-619.7	-651.4	-609.1	-639.4
icorr (mA.cm^{-2})	0.1467	0.0501	0.0137	0.1274
Rp (kΩ.cm^{2})	0.298	0.452	1.240	0.161
βa (mV)	95.7	84.0	75.4	79.7
-βc (mV)	227.7	268.0	156.2	231.4
EI_P (%)	-	65.84	90.66	13.15

To further explore the effect of DPL extract on the corrosion behavior of CS in 3.5% NaCl, Electrochemical Impedance Spectroscopy (EIS) tests were conducted. Figure 5 presents the Nyquist plot, while Table 5 summarizes the EIS parameters derived from fitting. Interestingly, the Nyquist plot shows a gradual increase in the semicircle diameter with higher DPL extract concentrations, notably up to 2.10^{-4} g.L^{-1}, indicating a reduction in the corrosion rate. This insight reinforces the potential of DPL extract as a corrosion inhibitor.
Moreover, with increasing the DPLE concentration, a noticeable decrease in Q_{dl} values has been observed. This trend can be explained by the adsorption of DPLE molecules on the metal surface, causing a decrease in the active metal surface area available for interaction with aggressive ions in the corrosive medium [32,33].

3.2.3. Surface morphology studies

In Figure 6A, the polished surface of carbon XC70 steel appears smooth and devoid of pits. However, the lines observed are attributed to the cutting marks during sample preparation. Figure 6B illustrates the scanning electron microscopy (SEM) image obtained for the steel samples after immersion for 24 hours in a corrosive solution without DPLE. The image shows a substantial transformation of the metal surface indicating a severe corrosion by oxidation in the saline medium in the absence of inhibitors.

The saline attack resulted in the development of several cracks and pits on the metal surface. The formation of iron-oxide is a direct consequence of the chemical interaction between the outer iron layer and the corrosive solution.

Figure 6C shows the scanning electron microscopy (SEM) image of the XC70 steel after immersion for 24 hours in a corrosive medium with 2.10^{-4} g.L^{-1} of DPLE. The metal surface has visibly undergone a slight corrosion in the presence of a moderate amount of DPLE in the saline medium, which reduced significantly the metal corrosion by the formation of a protective layer on the metal surface.

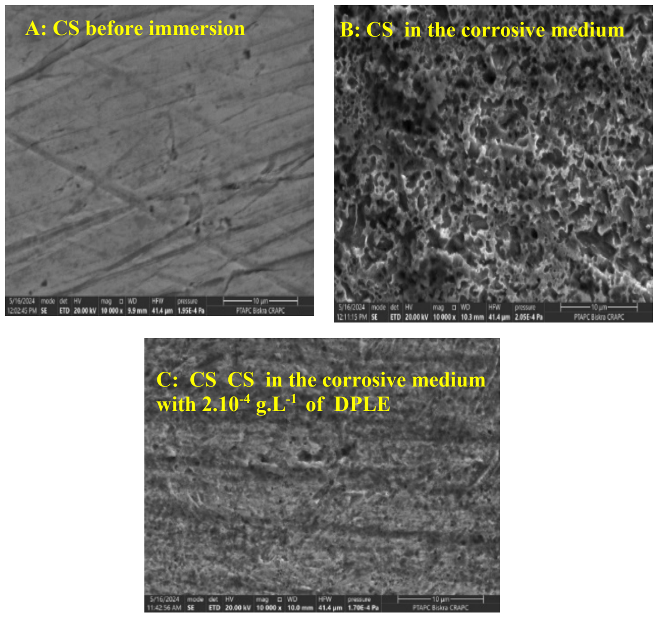

Figure 6. SEM of CS

Figure 7 illustrates AFM images of the CS after immersion for 24 hours in a corrosive medium, without and with 2.10^{-4} g.L^{-1} of DPLE. A 3D analysis of the CS surface immersed in the corrosive solution without DPLE (Fig. 7a) showed peaks and valleys with an average roughness of about 749.3 nm on the metal surface. With the addition of DPLE in the saline solution, the roughness is found to decrease to 317.8 nm (Fig. 7b), suggesting that the presence of 2.10^{-4} g.L^{-1} of DPLE formed a protective layer on the carbon XC70 steel surface.

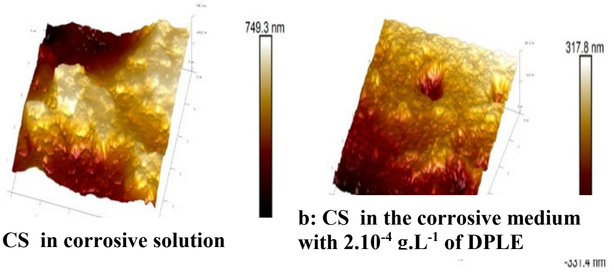

Figure 7. AFM of CS

4. CONCLUSION

The study findings led to the following conclusions:

- The structural characterization of the *Chamaerops humilis L.* leaf extract (DPLE) through GC-MS, phytochemical analysis and infrared spectrometry revealed the existence of compounds renowned for their antioxidant properties such as alkaloids, tannins, flavonoids, polyphenols. These substances combine to create a safeguarding shield against corrosion on the surface of the investigated carbon XC70 steel.
- In this work, electrochemical methods were employed to evaluate the corrosion inhibition efficacy of carbon XC70 steel containing various concentration dwarf palm leaf extract as inhibitor against a 3.5% NaCl solution attack. The Weight loss measurements and the electrochemical results showed that the extract under study as inhibitor showed a high effectiveness in fighting deterioration of the carbon XC70 steel in the saline solution.
- Finally, the dwarf palm leaf extract exhibited a satisfactory corrosion inhibitory effect on carbon steel, which is a promising process using environmentally friendly inhibitors.

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