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Cardiospermum halicacabum leaves extract as a green corrosion inhibitor for mild steel in simulated oil well water medium

K. Kavitha,[1,2] H. Benitasherine[2] and S. Rajendran[3,4]*

[1]PG and Research Department of Chemistry, National College (Autonomous), Tiruchirappalli-620001, Tamil Nadu, India
[2]PG and Research Department of Chemistry, Periyar E.V.R. College (Autonomous), Tiruchirappalli-620023, Tamil Nadu, India
[3]Corrosion Research Centre, Department of Chemistry, St. Antony's College of Arts and Sciences for Women, Dindigul-624005, Tamil Nadu, India
[4]Adjunct Professor, Pondicherry University, Puducherry, India
**E-mail: susairajendran@gmail.com*

Abstract

The green corrosion inhibition properties of an aqueous extract of *Cardiospermum halicacabum* leaves (CHL) were studied on mild steel (MS) in simulated oil well water (SOWW) medium by weight loss method and potentiodynamic polarization study. The active principle components are characterized by Fourier Transform Infrared Spectroscopy (FTIR). The surface morphology is examined with the help of scanning electron microscopy (SEM) and the surface roughness analysis is done by atomic force microscopy (AFM). Weight loss method reveals that as the concentration of inhibitor increases, the corrosion rate decreases and inhibition efficiency increases. A maximum corrosion inhibition efficiency of 99% is offered by the inhibitor system. Potentiodynamic polarization study shows that CHL acts as mixed type of inhibitor, controlling anodic reaction and cathodic reaction to an equal extend. This is confirmed by the fact that the shift in corrosion potential in presence of inhibitor is very small, that is, –13 mV *vs*. SCE. Protective film formation against corrosion was confirmed by Scanning electron microscopy (SEM) and Atomic force microscopy (AFM) studies. In presence of inhibitor a protective film is formed, which is found to be very smooth when compared with the uninhibited system. Root-mean-square roughness, average roughness and peak-to-valley height value for the protective film are found to be lower than those of unprotected metal surface. The results show that the CHL extract could serve as good green corrosion inhibitor for mild steel immersed in simulated oil well water. The outcome of the study may find application in petroleum industry.

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Keywords: *cardiospermum halicacabum leaves, corrosion inhibitor, mild steel, simulated oil well water, surface morphology.*

Introduction

Corrosion is a universal occurrence and prevention of corrosion becomes one of the challenging phenomena in the modern world. When a reactive metal or alloy is exposed to

environment, the gases and water present in air react gradually with the metal surface and form their corresponding oxides, carbonates, sulphates, *etc.* This eating up of a metal by the environment is called as corrosion [1]. Corrosion prevention should not be considered as a financial issue, but also one of the health and safety issues to the environment [2]. Due to high strength, ease of fabrication and low cost, iron and steel are widely used in the steel frame of buildings, machinery parts, cookware, pipeline, storage tanks for water, liquid chemicals and petroleum products, paints, water treatment plant, oil industries, biofuel, visually aesthetic metal gate, fencing *etc.* [3, 4].

A corrosion inhibitor is a substance when added in a small concentration to an environment reduces the corrosion rate of a metal exposed to that environment [5]. Corrosion inhibitors commonly used nowadays are toxic and harmful to the environment, which makes it difficult to handle and to dispose of them. Many of the inhibitors used that have good anti-corrosion properties have synthetic components in their composition, being toxic to humans and to the environment [6–9]. Natural corrosion inhibitors are renewable, non-toxic, readily available, economically viable and do not contain heavy metals [10]. The plant extract has been employed as effective inhibitors of corrosion due to their low cost, biodegradability, high availability and non-toxic nature [11, 12]. So, the study of plant extracts as corrosion inhibitors has received more attention due to environmental benefits. Several naturally occurring plant such as *Ocimum gratissimum* [13], Lignin extract of sun flower (*Tithonia diversifolia*) [14], turmeric powder [15], *Capsicum annum* fruit paste [16], human black hair extract [17], *Robinia pseudoacacia* leaves extract [18], leaves of purple Knight Hedge plant [19] and *Wihania somnifera* [20] were used as possible sources of green corrosion inhibitors by several researches. Suleiman et al have used the ethanolic extract of *Cardiospermum halicacabum* leaves for the investigation of corrosion of steel pipeline in 0.5 M HCl [21]. Deivanayagam and selvaraj have studied the ethanolic extract of *Cardiospermum halicacabum* leaves for the corrosion of Brass in 1.0 N HCl solution [22]. However, to the best of our knowledge the aqueous extract of *Cardiospermum halicacabum* plant has not been investigated in SOWW medium for its anticorrosion properties.

Cardiospermum halicacabum (Sapindaceae) (Figure 1) is a climber of about 2–4 in long, evergreen, branched with inflated fruits. They are distributed in tropical and subtropical regions of Africa, Asia and often consumed as green leafy vegetable in India. *C. halicacabum* is commonly known as Balloon vine, heart pea (England), Parol-paralon (Philippines), Jia Hu Gua (China) and Mudakathan Keerai (Tamil Nadu, India). *C. halicacabum* is used in Indian traditional medicine system for the treatment of rheumatism, lumbago, cough, hyperthermia, nervous diseases, stiffness of limbs and snake bites [23]. The aqueous extract of *Cardiospermum halicacabum* leaves contain main active principle constituents such as tricyclo[5.4.3.0(1,8)]tetradecan-6-one, 4-ethenyl and apigenin sulfate identified from GC-MS study [24]. The structures of the molecules are shown in Figure 2.

In the present study, the corrosion inhibitive properties of the aqueous extract of *Cardiospermum halicacabum* leaves in controlling corrosion of mild steel in SOWW

solution have been investigated by weight loss and potentiodynamic polarization technique. The CHL extract of active principle constituents have been characterized by FTIR, with the formation of protective film formation against corrosion. The morphology of the mild steel surface was examined by SEM and AFM.

Figure 1. Cardiospermum halicacabum leaves.

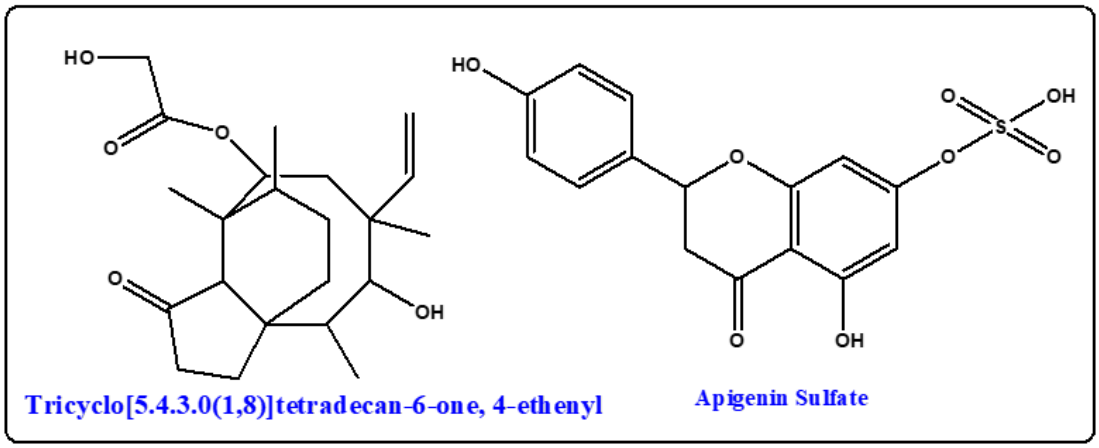

Figure 2. The structure of tricyclo[5.4.3.0(1,8)]tetradecan-6-one, 4-ethenyl and apigenin sulfate.

Experimental section

Preparation of inhibitor

The aqueous extract of *Cardiospermum halicacabum* leaves (CHL) was prepared by the method of soxhlet extraction. About 100 g of powdered plant of *Cardiospermum halicacabum* leaves was uniformly packed into thimble and extracted with 1000 ml of double distilled water. The process of extraction continued till the solvent in siphon tube of the extractor became colourless. After the process of extraction, the extract was kept

overnight for cooling and made up to 1000 ml with the same double distilled water to get 10% (*w*/*v*) extract.

Preparation of simulated oil well water (SOWW)

In 100 mL of double distilled water, sodium chloride (3.5 g), calcium chloride (0.305 g) and magnesium chloride (0.186 g) are added. Just before experiment add 0.067 g sodium sulfide and 0.4 mL of concentrated hydrochloric acid to generate hydrogen sulfide gas to form a simulated oil well water containing 100 ppm of H_2S [25].

Preparation of mild steel (MS)

Mild steel specimens (S = 0.0267%, P = 0.06%, Mn = 0.4%, C = 0.1% and the rest iron) of dimensions $1.0\times4.0\times0.2$ cm^3 were polished to a mirror finish and degreased with acetone.

Weight loss method

Mild steel specimens in triplicate were immersed in 100 ml of the simulated oil well water containing various concentrations of the inhibitor (aqueous extract of *Cardiospermum halicacabum*leaves) for a period of one day. The weight of the specimens before and after immersion was determined using a Shimadzu balance, model AY62. The corrosion products were cleaned with Clarke's solution [26]. The corrosion rates were calculated using the following equation [27].

$$\text{Corrosion} = W/AT$$

Where:

$$W = \text{loss in weight } (\text{mg})$$

$$A = \text{surface area of the specimen } (\text{dm}^2)$$

$$T = \text{period of immersion } (\text{days})$$

The corrosion rate is expressed in mdd units [mdd = mg/(dm^2)·(day)]. The inhibition efficiency was calculated using the relation:

$$\text{Inhibition efficiency} = \left[(CR_1 - CR_2)/CR_1\right]\cdot 100\%$$

Where:

$$CR_1 = \text{corrosion rate in the absence of inhibitor}$$

$$CR_2 = \text{corrosion rate in the presence of inhibitor}$$

Electrochemical studies: Polarization study

In the present work, corrosion resistance of mild steel immersed in various test solutions were measured by potentiodynamic polarization technique. The experiments were done at room temperature. Polarization studies were carried out in a CHI– electrochemical work station with impedance model 660 A. It was provided with i_R compensation facility. A three electrode cell assembly was used. Mild steel was used as working electrode, platinum as counter electrode and saturated calomel electrode (SCE) as reference electrode. From polarization study, corrosion parameters such as corrosion potential (E_{corr}), corrosion current (I_{corr}), Tafel slopes anodic = b_a and cathodic = b_c and linear polarization resistance (LPR) value were calculated [28].

FTIR spectra

FTIR spectra were recorded in a Perkin – Elmer "Spectrum Two" spectrophotometer. The film was carefully removed, mixed thoroughly with KBr, made in to pellets and FTIR spectra were recorded.

Surface characterization study

The mild steel specimens were immersed in various test solutions for a period of one day. After one day the specimens were taken out and dried. The nature of the film formed on the metal surface was analyzed by surface characterization studies such as scanning electron microscopy (SEM) and atomic force microscopy (AFM).

Scanning electron microscopy (SEM)

The mild steel specimens immersed in various test solutions for one day were taken out, rinsed with double distilled water, dried and subjected to the surface examination. The surface morphology measurements of the mild steel surface were carried out by scanning electron microscopy (SEM) using CAREL ZEISS make model EVO-18.

Atomic force microscopy (AFM)

The mild steel specimens immersed in various test solutions for one day were taken out, rinsed with double distilled water, dried and subjected to the surface examination. The surface morphology measurements of the mild steel surface were carried out by atomic force microscopy (AFM) using SPM Veeco diInnova connected with the software version 7.00 V and the scan rate was 0.7 Hz.

Results and Discussion

Pipelines made of MS are used to carry oil well water in petroleum industry. These pipes may undergo inner corrosion. To prevent corrosion, corrosion inhibitors are used. The corrosion resistance of MS in SOWW, has been evaluated by polarization study. Aqueous extract of *Cardiospermum halicacabum* leaves (CHL) has been used as corrosion inhibitor.

Weight loss method

Weight loss method is a non-electrochemical technique to determine the rate of corrosion and inhibition efficiency. The data obtained from weight loss method of mild steel corrosion in SOWW without and with CHL of various concentrations for one day are shown in Table 1. From the table, it is evident that as the concentration of inhibitor increases, inhibition efficiency increases and rate of corrosion decreases. The maximum inhibition efficiency of 99% was attained for mild steel when immersed in 10% *v*/*v* of extract concentration. Formation of a thin film of adsorbed inhibitor molecules can be proposed to explain this observation [29, 30]. This adsorbed film acts as a barrier to the corrosion process leading to higher inhibition efficiency.

Table 1. The corrosion rate of mild steel and the efficiency of the inhibitor in different concentrations in SOWW medium.

Inhibitor (CHL % *v*/*v*)	**Corrosion rate (CR) mdd**	***IE*%**
0	14.55	–
2	2.91	80
4	1.89	87
6	1.16	92
8	0.73	95
10	0.15	99

Electrochemical study. Analysis of polarization study

Polarization study has been extensively used in the corrosion inhibition studies. In the presence of inhibitors, linear polarization resistance (LPR) value increases and corrosion current (I_{corr}) value decreases. The polarization curves of MS electrode immersed in SOWW medium in the absence and presence of inhibitor (CHL) system are shown in Figure 3. Electrochemical parameters such as corrosion potential (E_{corr}), Tafel slopes (b_c, b_a), linear polarization resistance (LPR) values and corrosion current (I_{corr}) values are given in Table 2. When mild steel was immersed in SOWW the corrosion potential was –831 mV *vs*. SCE. When 10% of *Cardiospermum halicacabum* leaves extract was added to the above system the corrosion potential shifted to the anodic side –818 mV *vs*. SCE. This indicates that the anodic reaction is controlled predominantly. However, the shift is within ±85 mV [31]. This indicates that the inhibitor behaves as a mixed type inhibitor controlling both anodic reaction and cathodic reaction to an equal extent by forming a protective film. Further, the LPR value increases from 501 Ohm·cm^2 to 16316 Ohm·cm^2. The corrosion current decreases from $7.688 \cdot 10^{-5}$A/cm^2 to $1.281 \cdot 10^{-6}$ A/cm^2. Hence, polarization study confirms the formation of a protective film on the metal surface. The inhibition efficiency calculated from corrosion

current was found to be 98.33%. The above inferences indicate that the corrosion rate of the mild steel reduced through the formation adsorbed protective film on the metal surface.

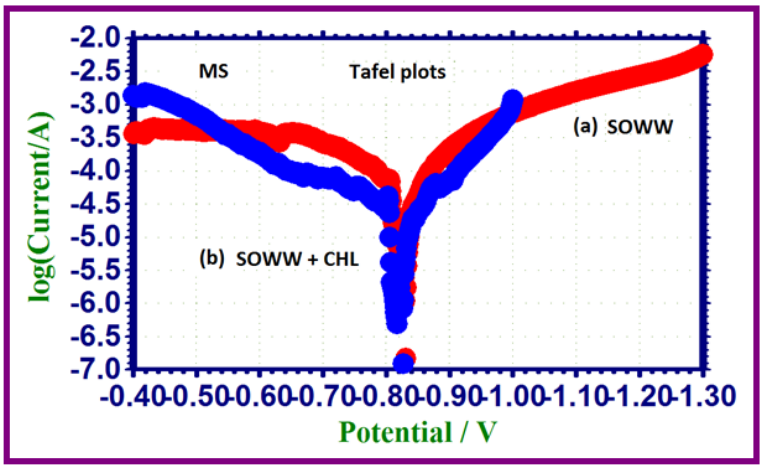

Figure 3. Polarization curves of mild steel immersed in various test solutions (a) SOWW, (b) SOWW + inhibitor (10% CHL).

Table 2. Tafel polarization parameters of mild steel in SOWW with different concentrations of CHL.

System	E_{corr}, mV *vs.* SCE	b_c, mV/decade	b_a, mV/decade	*LPR*, Ohm·cm^2	I_{corr}, A/cm^2	*IE*%
SOWW	–831	150	216	501	$7.688 \cdot 10^{-5}$	–
SOWW + 10% CHL	–818	108	87	16316	$1.281 \cdot 10^{-6}$	98.33

FTIR spectra

FTIR spectra have been used to analyse the protective film formed on the metal surface. A few drops of an aqueous extract of *Cardiospermum halicacabum* leaves were dried on a glass plate. A solid mass was obtained. It was blended with KBr and converted into pellet. Its FTIR spectral pattern is recorded and shown in Figure 4. A broad band appears at 3428.79 cm^{-1} is attributed to –OH group. The band observed at 2930.80 cm^{-1} represents the aliphatic C–H stretching. The Peak at 1633.71 cm^{-1} shows the presence of C=O and aromatic ring (C=C) group. The peak at 1406.71 cm^{-1} has been assigned to S=O stretching frequency. The band 1249.05 cm^{-1} indicates the presence of C–O stretching group. The peak at 1074.24 cm^{-1} is assigned to C–O–C stretching vibration. The C–H out of plane bending (oop) absorbs at 618.11 cm^{-1}. Thus the structure of main active principle component of *Cardiospermum halicacabum* leaves extract namely tricyclo[5.4.3.0(1,8)]tetradecan-6-one, 4-ethenyl and apigenin sulfate is confirmed by FTIR spectrum.

The FTIR spectrum of the protective layer formed on the mild steel surface after immersion in the solution containing SOWW with 10% *v*/*v* of inhibitor (CHL) solution is shown in Figure 5. The slight shift at 3428.79 cm^{-1} to 3427.54 cm^{-1} can be attributed to the

presence of –OH stretching. The shift from 2930.80 cm^{-1} to 2924.14 cm^{-1} indicates the presence of C–H bond. The peak shifted from 1633.71 cm^{-1} to 1631.05 cm^{-1} indicates the presence of C=O and C=C aromatic ring group. The S=O stretching frequency has shifted from 1406.71 cm^{-1} to 1383.35 cm^{-1}. The frequency 1249.05 cm^{-1} for C–O group is shifted to 1117.62 cm^{-1}. The C–O–C stretching frequency has shifted from 1074.24 cm^{-1} to 1021.62 cm^{-1}. The peak C–H "oop" bending shifted from 618.11 cm^{-1} to 694.82 cm^{-1}. Thus it is observed that tricyclo[5.4.3.0(1,8)]tetradecan-6-one, 4-ethenyl and apigenin sulfate has coordinated with Fe^{2+} through oxygen atom and sulfur atom of tricyclo-[5.4.3.0(1,8)]tetradecan-6-one, 4-ethenyl or apigenin sulfate. It is confirmed that the protective film is Fe^{2+}–Tricyclo[5.4.3.0(1,8)]tetradecan-6-one, 4-ethenyl or Fe^{2+}–apigenin sulfate complex formed on the metal surface [22, 32, 33].

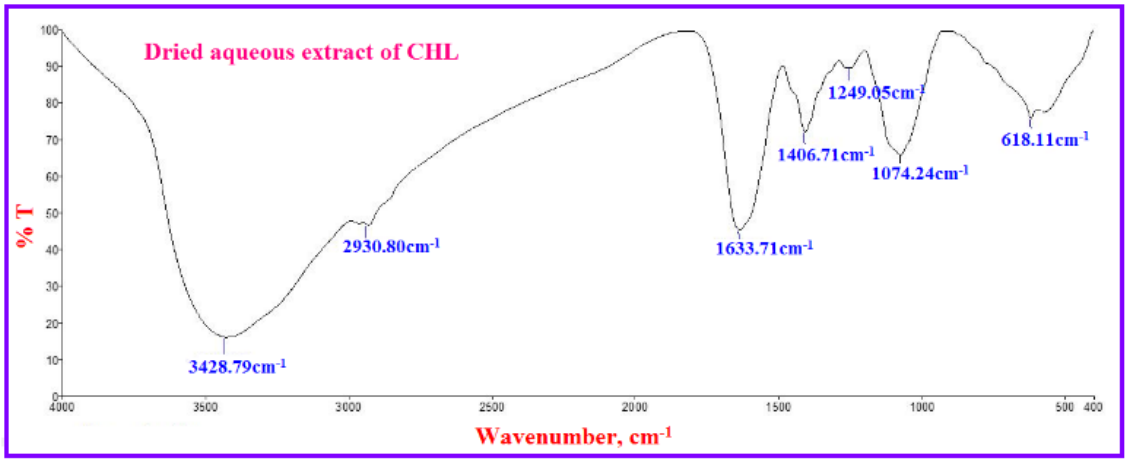

Figure 4. FTIR spectrum of pure aqueous extract of CHL evaporated to dryness.

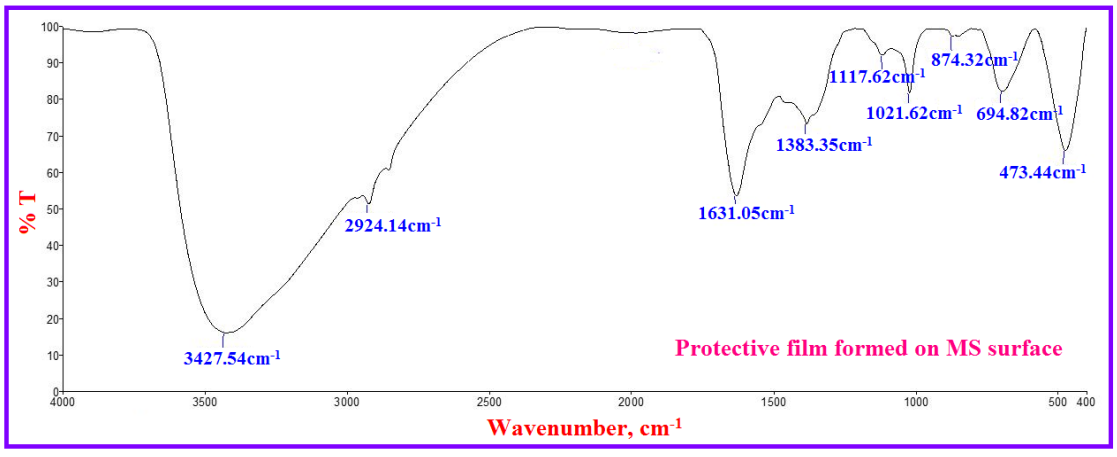

Figure 5. FTIR spectrum of protective film formed on the mild steel surface during the immersion in SOWW containing inhibitor (CHL).

Analysis of SEM

Scanning electron microscopy (SEM) was used to examine the surface morphology of the mild steel species. The SEM images of magnification (3000x) of mild steel specimen and mild steel specimen immersed in SOWW for one day in the absence and presence of inhibitor system are shown in Figure 6 as images (a, b, c) respectively.

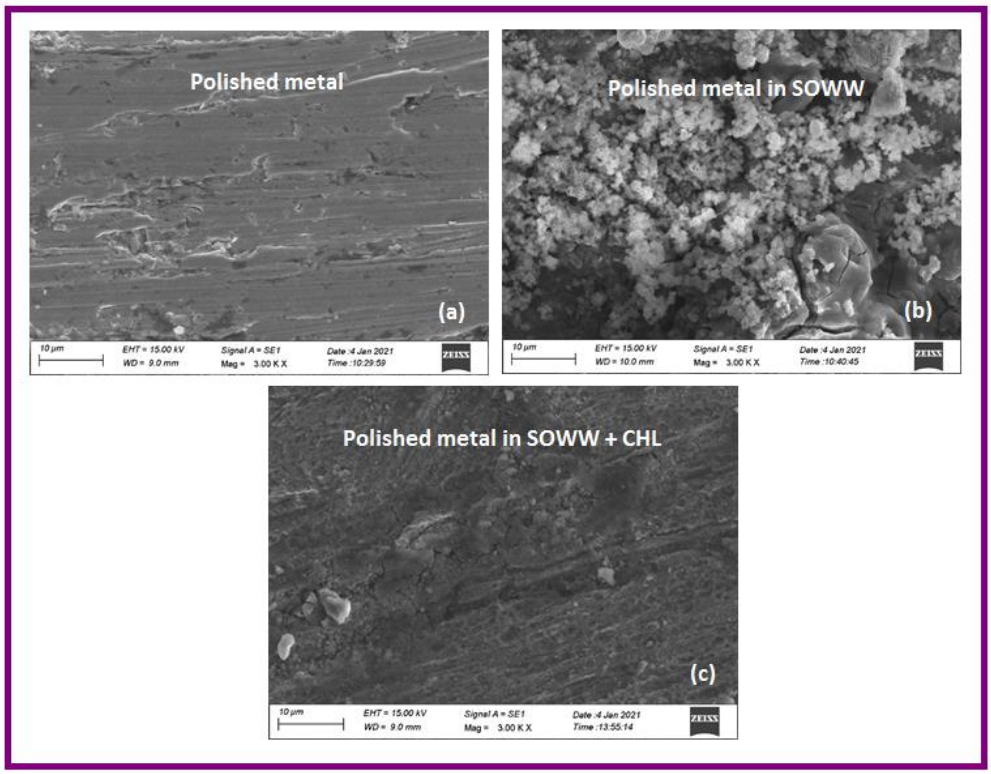

Figure 6. SEM images of (a) polished mild steel (b) mild steel immersed in SOWW (c) mild steel immersed in SOWW containing inhibitor (CHL).

The SEM micrograph of polished mild steel surface (control) in Figure 6a shows the smooth surface of the metal. The surface is considerably quite good and even. The SEM micrograph of mild steel surface immersed in SOWW in Figure 6b shows the roughness of the metal surface which indicates the corrosion of mild steel in SOWW. The SEM image of the mild steel surface immersed in SOWW containing the inhibitor CHL is shown in Figure 6c. A smooth surface is noticed. This indicates that a protective film is formed by the adsorption of the inhibitor on the metal surface, thus preventing the corrosion. The images confirm that CHL inhibitor is effective in controlling corrosion by hindering the reactive active sites on the mild steel surface [34, 35].

Atomic force microscopy

Atomic force microscopy is a powerful technique to investigate the surface morphology at nano- to micro-scale and has become a new choice to study the influence of inhibitor on the generation and the progress of the corrosion at the metal/solution interface [36–39].

The two-dimensional (2D) AFM cross-sectional and three dimensional (3D) AFM morphologies for polished mild steel surface, mild steel immersed in SOWW (blank) and mild steel immersed in SOWW containing 10% aqueous extract of inhibitor CHL are shown in Figures 7 and 8 respectively. Root-mean-square roughness, average roughness and peak-to-valley height values were measured. AFM image analysis was performed to obtain the average roughness, R_a (the average deviation of all points roughness profile from a mean line over the evaluation length), root-mean-square roughness, R_q (the average of the measured height deviations taken within the evaluation length and measured from the mean line) and the maximum peak-to-valley ($P–V$) height values (largest single peak-to-valley height in five adjoining sampling heights) [37]. The parameters R_q, R_a and $P–V$ height from the AFM images of metal surfaces are given in Table 3 for the polished mild steel and that after immersion in the absence and presence of 10% *v*/*v* aqueous extract of inhibitor CHL.

Figures 7a and 8a show the surface topography of polished mild steel surface. The value of R_q, R_a and $P–V$ height for polished mild steel surface are 116.89 nm, 98.65 nm and 477.84 nm respectively. The roughness observed on polished mild steel surface is due to atmospheric corrosion. Figures 7b and 8b show the corroded metal surface with pits are noticed in mild steel immersed in SOWW medium (blank). The R_q, R_a and $P–V$ height values are significantly very high. It indicates the mild steel surface becomes rougher in SOWW medium, without the inhibitor. Figures 7c and 8c show the mild steel surface after immersion in SOWW containing 10% *v*/*v* of inhibitor CHL. The R_q, R_a and $P–V$ height values for the inhibited mild steel surface (with protective film) are 153.84 nm, 130.96 nm and 628.99 nm respectively. The values are higher than that of the polished mild steel and lower than that of the corroded mild steel surface. These parameters confirm that the surface is smoother. The smoothness of the surface is due to the formation of a protective film of Fe^{2+}–*Cardiospermum halicacabum* complex thereby inhibiting the corrosion of mild steel [40].

Table 3. AFM parameters of mild steel surface in the presence and absence of inhibitor (CHL) system.

Sample	RMS (R_q) Roughness (nm)	Average (R_a) Roughness (nm)	Maximum peak-to-valley height (nm)
Polished MS	116.89	98.65	477.84
MS immersed in SOWW	623.84	456.15	2901.5
MS immersed in SOWW and 10% *v*/*v* of CHL extract	153.84	130.96	628.99

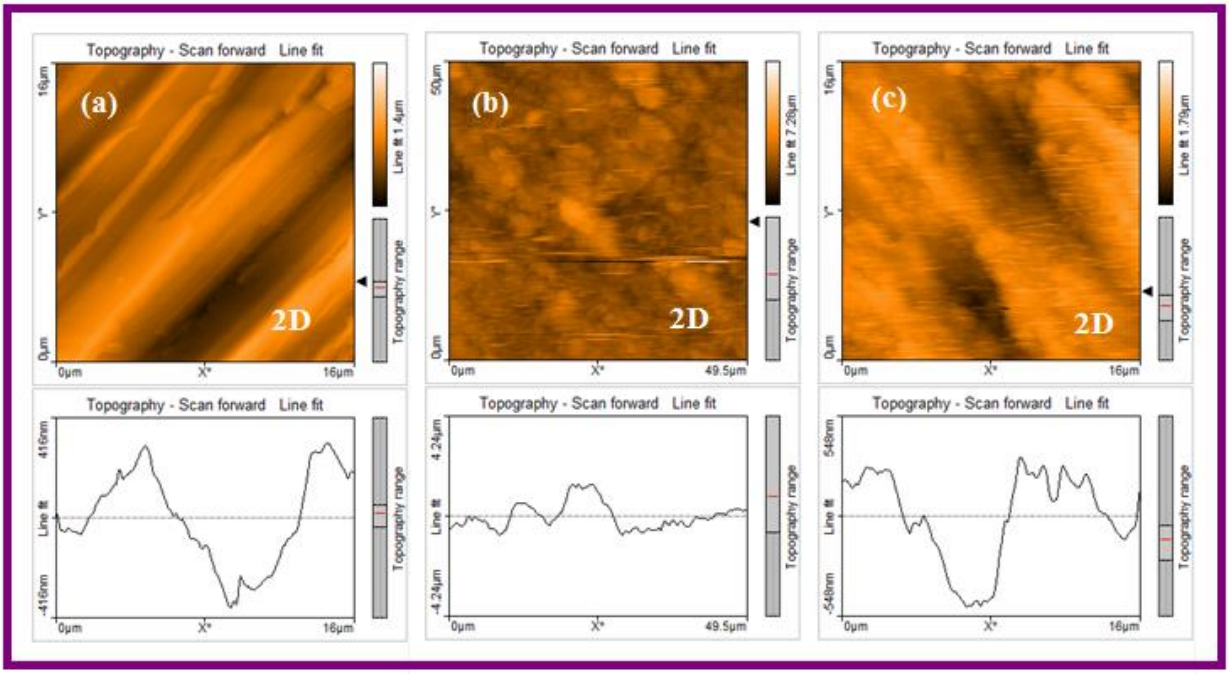

Figure 7. Two-dimensional AFM images (a) polished MS; (b) MS immersed in SOWW; (c) MS immersed in SOWW containing inhibitor (CHL) system.

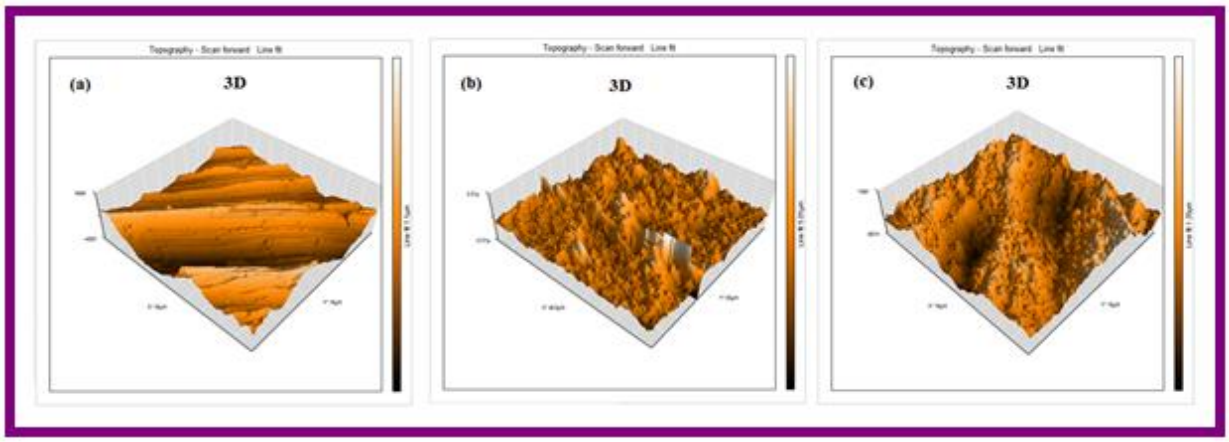

Figure 8. Three-dimensional AFM images of the surface (a) Polished MS; (b) MS immersed in SOWW; (c) MS immersed in SOWW containing inhibitor (CHL) system.

Conclusions

The corrosion inhibition efficiency (*IE*%) of an aqueous extract of *Cardiospermum halicacabum* leaves system in controlling corrosion of mild steel in SOWW has been evaluated by weight loss method. The study leads to the following conclusions.

- Weight loss study reveals that the formulation consisting of 10% *v*/*v* of CHL has 99% inhibition efficiency in controlling corrosion of mild steel immersed in SOWW.

- FTIR results reveal that a protective film is formed on the metal surface consisting of Fe^{2+}–CHL (active principle) complex.
- Electrochemical study such as polarization technique has been employed to investigate the mechanistic aspects of the corrosion inhibition. The study confirms the formation of protective film formed on the metal surface.
- The aqueous extract of CHL behaves as mixed type corrosion inhibitor.
- The SEM and AFM analysis showed that the inhibition of mild steel occurred by the development of inhibitive film on the metal surface.

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