Aqueous extract of Cassia fistula flower as green corrosion inhibitor for mild steel in simulated oil well water medium

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Aqueous extract of *Cassia fistula* flower as green corrosion inhibitor for mild steel in simulated oil well water medium

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Abstract

The inhibition efficiency of aqueous extract of *Cassia fistula* flower (CFF) in controlling corrosion of mild steel in simulated oil well water (SOWW) medium has been evaluated by weight loss method and electrochemical measurement (potentiodynamic polarization study). The extract has been characterized by Fourier Transform Infrared Spectroscopy (FTIR). The active ingredients present in the CFF have been identified by Gas Chromatography Mass Spectroscopy (GCMS). A maximum inhibition efficiency of 95% has been obtained at 10% of CFF extract. The polarization study reveals that this formulation acts as a barrier film controlling the cathodic reaction predominantly. The corrosion potential is shifted from –831 to –875 mV *vs.* SCE. The inhibitor system functions as mixed type of inhibitor because the shift in corrosion potential is less than 85 mV. The linear polarization resistance value increases from 501 $Ohm \cdot cm^2$ to 1335 $Ohm \cdot cm^2$. The corrosion current decreases from $7.688 \cdot 10^{-5}$ A/cm^2 to $2.593 \cdot 10^{-5}$ A/cm^2. These factors confirm that the *Cassia fistula* flower extract controls the corrosion of mild steel in SOWW. The surface morphology has been examined with the help of scanning electron microscopy (SEM) and atomic force microscopy (AFM). It is observed that in presence of inhibitor the surface of the corroded metal becomes smoother. The findings have potential application in petroleum industry. The inhibitor extract can be added along with the simulated oil well water in pipeline made of mild steel.

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Keywords: *Cassia fistula flower, corrosion inhibitor, mild steel, simulated oil well water, electrochemical studies.*

Introduction

Petroleum industry is a very important industry because many transporting vehicles depend on petroleum. Many pipelines are used to transport oil well water to refineries. These pipelines are made of materials such as mild steel, various stainless steels such as L80 alloy *etc.* When oil well water is transported through these pipelines, they may undergo corrosion. The main mechanism for internal corrosion of pipelines is aqueous corrosion caused by soluble corrosive gas, such as carbon dioxide, hydrogen sulfide or oxygen and corrosion is influenced by microorganisms. To prevent corrosion usually inhibitors are added. Many research papers have been published on the use of corrosion inhibitors to control corrosion of pipelines carrying oil well water.

Several inhibitors have been used for this purpose. Anusuya and Devi Meenakshi have used trisodium phosphate to control corrosion of pipe lines carrying simulated oil well water [1]. Pinnock *et al.* have investigated the use of carbon steel ball bearings to determine the effect of biocides and corrosion inhibitors on microbiologically influenced corrosion under flow conditions [2]. Synthesis and characterization of decyl phosphonic acid, and its applications in emulsion polymerization and anti-corrosion coating have been reported by Phan *et al.* [3]. Novel Gemini cationic surfactants as anti-corrosion for X-65 steel dissolution in oilfield produced water under sweet conditions have been used by Migahed *et al.* [4]. A new mixture of phosphonate scale inhibitors to control scales during water flooding has been proposed by Khormali *et al.* [5]. Mainier *et al.* have reported a study on calcium carbonate scale in steam generator in enhanced oil recovery [6]. Gómez *et al.* have evaluated the use of corrosion inhibitors in simulating conditions of operations [7]. Migahed *et al.* have found that a novel ionic liquid compound can act as sweet corrosion inhibitor for X-65 carbon tubing steel [8]. Ethanol extract of *Cardiospermum halicacabum* leaf has been used to control corrosion of steel pipelines in acidic environment by Suleiman *et al.* [9]. Riaz Ahamed *et al.* have used an aqueous extract of *Pedalium murex L.* Leaves to control corrosion of mild steel in acid medium [10]. Devi *et al.* have utilized an aqueous extract of henna leaves (*Lawsoniainermis*) as corrosion inhibitor [11]. An aqueous extract of *Bauhinia Blakeana* leaves has been used by Geetha *et al.* to control corrosion of mild steel in bore well water [12]. *Leucaena leucocephala* leaves extract has been used by Ikhmal *et al.* to control corrosion of mild steel in sea water [13]. Prabha *et al.* have investigated the use of an aqueous extract of *Andrographis paniculata* in controlling corrosion of mild steel in simulated oil well water [14]. Mahalakshmi *et al.* have used an aqueous extract of turmeric powder to mitigate corrosion of mild steel in sea water [15].

Cassia fistula, commonly known as golden shower, purging cassia, Indian laburnum or pudding-pipe tree, is a flowering plant in the family *Fabaceae*. The species is native to the Indian subcontinent and adjacent regions of Southeast Asia, from southern Pakistan through India and Sri Lanka to Bangladesh, Myanmar and Thailand. It is a popular ornamental plant and is also used in herbal medicine. It is an ornamental tree with long and cylindrical fruits (Figure 1). It is also used to cure burns, constipation, convulsions, diarrhea, dysuria and epilepsy. Ayurvedic medicines recognize it as carminative, laxative and to cure leprosy, skin diseases and syphilis [16, 17].

In the present study extract of CFF has been chosen for the inhibition of corrosion of mild steel pipelines carrying simulated oil well water (SOWW). The presence of heteroatoms present in CFF has been identified by GC-MS. The corrosion inhibition efficiency has been tested via weight loss method and potentiodynamic polarization study. The protective film formed on the mild steel surface in the inhibitor system has been analysed by using surface analysis techniques such as SEM and AFM. The formation of metal inhibitor complex on the mild steel surface has been confirmed by FTIR spectroscopy.

Figure 1. *Cassia fistula* flower.

Experimental Section

Preparation of inhibitor

The aqueous extract of *Cassia fistula* flower (CFF) was prepared by the method of Soxhlet extraction. About 100 g of powdered plant of *Cassia fistula* flower was uniformly packed into the thimble and extracted with 1000 ml of double distilled water. The process of extraction continued till the solvent in siphon tube of the extractor becomes colorless. 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, 0.067 g sodium sulfide and 0.4 ml of concentrated hydrochloric acid are added to generate hydrogen sulfide gas to form a simulated oil well water containing 100 ppm of H_2S [18].

Preparation of mild steel (MS)

Mild steel specimens (0.0267% S, 0.06% P, 0.4% Mn, 0.1% C and the rest iron) of dimensions 1.0×4.0×0.2 cm were polished to a mirror finish and degreased with acetone.

Gas Chromatography – Mass Spectroscopy analysis of CFF

GC-MS is one of the advanced techniques used to identify the components of volatile matter, long chain and branched chain hydrocarbons, alcohols, acids, esters etc. GC-MS analysis was done on a Shimadzu 2010 plus comprising a AOC-20i auto sampler and gas chromatography interfaced to a mass spectrometer instrument. Interpretation on GC-MS was conducted using the database of National Institute Standard and Technology (NIST) having more than 62,000 patterns. The spectrum of the unknown component was compared with the spectrum of the known components stored in the NIST library. The name, molecular weight and structure of the components of the test materials were ascertained [19].

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 *Cassia fistula* flower) 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 [20]. The corrosion rate was calculated using the following equation [21].

$$\text{Corrosion rate} = \frac{W}{AT}$$

where: W is the loss in weight (mg); A is the surface area of the specimen (dm^2); T is the period of immersion (days)

The corrosion rate is expressed in mdd units [$\text{mdd} = \text{mg}/(\text{dm}^2 \cdot \text{day})$]. The inhibition efficiency was calculated using the relation.

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

where: CR_1 is the corrosion rate in the absence of inhibitor; CR_2 is the corrosion rate in the presence of inhibitor.

Electrochemical studies: Polarization study

In the present work, corrosion resistance of immersed mild steel in various test solutions were measured by potentiodynamic polarization study. The experiments were done at room temperature. Polarization studies were carried out in a CHI– electrochemical work station with impedance model 660A. It was provided with iR 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 have been calculated [22].

FTIR spectra

FTIR spectra were recorded in a Perkin-Elmer "Spectrum Two" spectrophotometer. The film was carefully removed, mixed thoroughly with KBr and 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 di Innova connected with the software version V7.00 and the scan rate of 0.7 Hz.

Results and discussion

An aqueous extract of *Cassia fistula* flower (CFF) has been used to control the corrosion of mild steel in presence of simulated oil well water (SOWW). The findings will be useful in petroleum technology. The inhibitor may be added to oil well water carried by pipelines made of mild steel. The inhibition efficiency of the inhibitor system was evaluated by weight loss method. The mechanistic aspects were studied by polarization study.

Gas Chromatography – Mass Spectroscopy analysis of CFF

The GC separated compounds are identified from the recorded mass spectra by comparison with the spectra from the database of National Institute of Standard Technology (NIST) library. The GC-MS chromatogram of the aqueous extract of *Cassia fistula* flower has 20 peaks indicating the presence of 20 chemical constituents (Figure 2). The 20 active constituents with their retention time (*RT*), molecular formula, molecular weight (*MW*) and peak area (%) in the aqueous extract of *Cassia fistula* flower are presented in Table 1. On comparison of the mass spectra of the constituents with the NIST library, the 3 predominant constituents were characterized and identified. The structure and nature of the compounds are presented in Table 2.

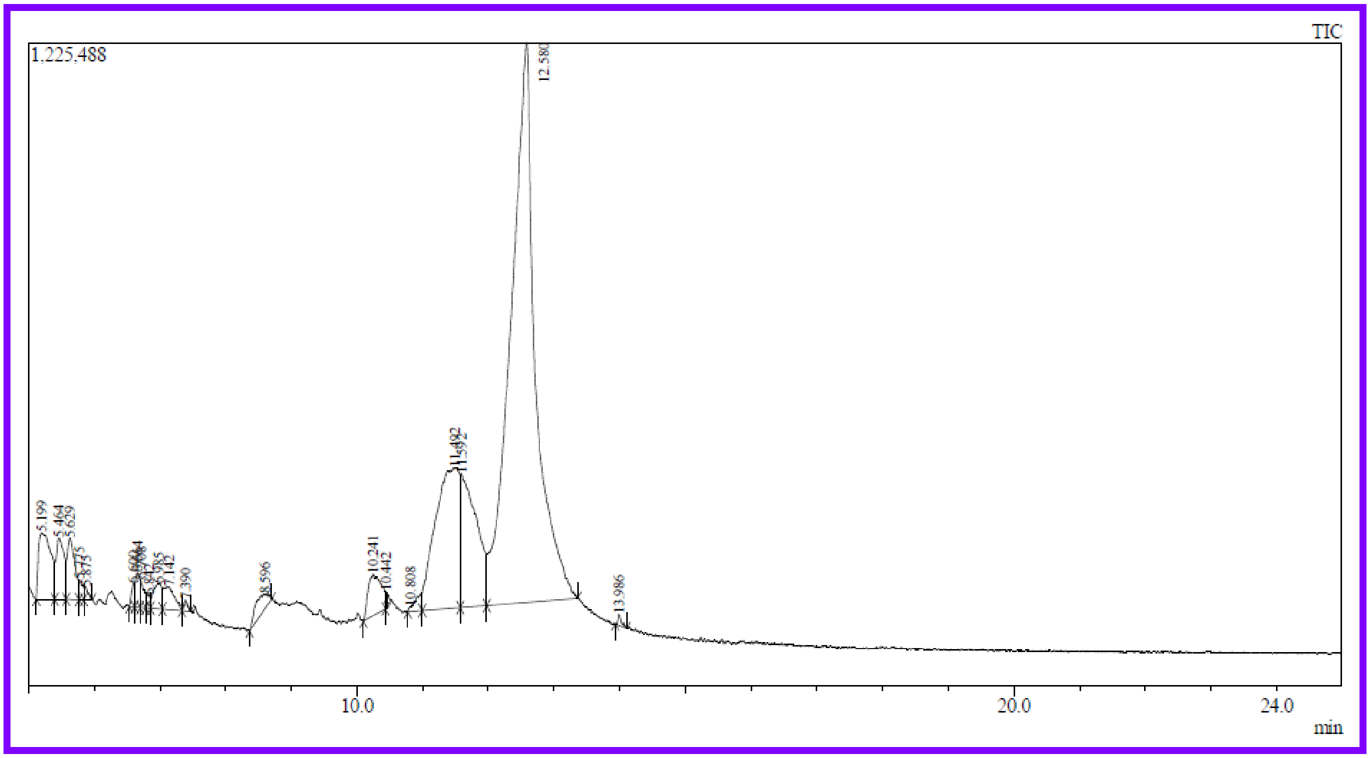


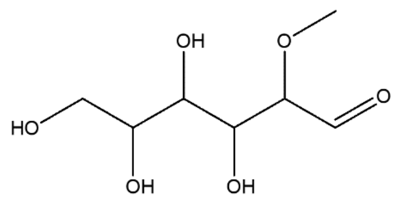
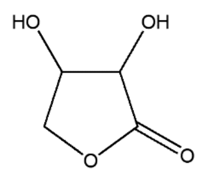
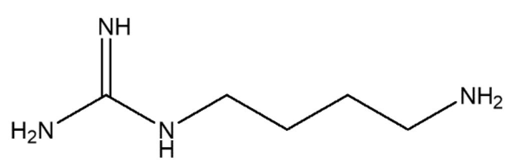
Figure 2. GC-MS spectrum for *Cassia fistula* flower.

Table 1. Phytochemical constituents identified in the aqueous extract of *Cassia fistula* flower by GC-MS.

S. No.	Name of the compound	Molecular Formula	Molecular Weight	Peak Area %
1	2-Furanone, 3,4-dihydroxytetrahydro	$C_4H_6O_4$	118	3.63
2	4-Hepten-3-one, 4-methyl-	$C_8H_{14}O$	126	2.13
3	1,2,3-Propanetriol, diacetate	$C_7H_{12}O_5$	176	1.97
4	Triethylsilanol	$C_6H_{16}OSi$	132	0.28
5	*o*-Methylisourea	$C_2H_6N_2O$	74	0.22
6	D-Erythro-Pentose, 2-deoxy-	$C_5H_{10}O_4$	134	0.31
7	1,2-Benzenedicarboxylic acid	$C_8H_6O_4$	166	0.63
8	2,4-Dimethoxy-2-methylbutane	$C_7H_{16}O_2$	132	0.41
9	Pantolactone	$C_6H_{10}O_3$	130	0.22
10	Butanal, 3-hydroxy-	$C_4H_8O_2$	88	0.92
11	Cyclohexene, 1-methyl-	C_7H_{12}	96	1.09
12	6-Hepten-3-ol	$C_7H_{14}O$	114	0.19

S. No.	Name of the compound	Molecular Formula	Molecular Weight	Peak Area %
13	Guanosine	$C_{10}H_{13}N_5O_5$	283	1.03
14	α-*D*-Glucopyranoside, methyl	$C_7H_{14}O_6$	194	2.34
15	Silane, (3-chloropropyl)trimethyl-	$C_6H_{15}ClSi$	150	0.12
16	Oxazolidine, 2-ethyl-2-methyl-	$C_6H_{13}NO$	115	0.56
17	2-Furanone, 3,4-dihydroxytetrahydro	$C_4H_6O_4$	118	14.65
18	(4-Aminobutyl)guanidine	$C_5H_{14}N_4$	130	9.27
19	Mome Inositol	$C_7H_{14}O_6$	194	59.86
20	Hexadecanoic acid	$C_{16}H_{32}O_2$	256	0.16

Table 2. The structure and nature of the predominant compounds.

S. No.	Name of the compound	Structure	Nature
1	Mome Inositol		Alcoholic compound
2	2-Furanone, 3,4-dihydroxytetrahydro (3,4-dihydroxydihydro-2(3*H*)-furanone)		Lactone compound
3	(4-Aminobutyl)guanidine (Agmatine)		Amine compound

Weight loss method

The corrosion rate and the inhibition efficiency of MS in SOWW medium in different concentrations of aqueous extract of *Cassia fistula* flower (CFF) have been calculated by weight loss method [23–28]. The values are given in Table 3. It is noticed from the Table 3 that as the concentration of inhibitor solution (CFF) increases, the corrosion rate decreases and the corrosion inhibition efficiency increases. A maximum corrosion inhibition efficiency of 95% is achieved. The increase in inhibition can be explained by the fact that when inhibitor concentration increases, more inhibitor molecules (active principle components in CFF) are adsorbed on the metal surface. Hence inhibition efficiency increases.

Table 3. The corrosion rate and the inhibition efficiency of MS in SOWW medium in different concentrations of inhibitor (CFF).

Inhibitor (CFF % *v/v*)	**Corrosion rate (*CR*) mdd**	***IE*%**
0	14.55	–
2	2.47	83
4	2.18	85
6	1.75	88
8	1.46	90
10	0.73	95

Electrochemical study: Analysis of polarization study

Corrosion parameters derived from polarization study, namely corrosion potential (E_{corr}), Tafel slope (b_c, b_a), linear polarization resistance (*LPR*) values and corrosion current (I_{corr}) values are given in Table 4. The polarization curves of mild steel (MS) immersed in SOWW in the absence and presence of inhibitor (CFF) system are shown in Figure 3.

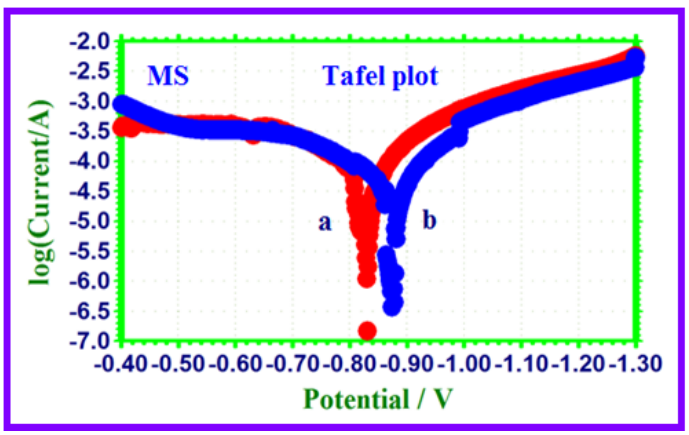


Figure 3. Polarization curves of mild steel immersed in various test solutions: (a) SOWW; (b) SOWW + inhibitor (CFF).

It is observed from the Table 4 that when mild steel is immersed in SOWW, the corrosion potential is –831 mV *vs.* SCE. The *LPR* value is 501 Ohm·cm^2 and corrosion current value is $7.688 \cdot 10^{-5}$ A/cm^2. It is inferred from the Table 4, that in the presence of inhibitor, the corrosion potential is shifted from –831 to –875 mV *vs.* SCE. An inhibitor can be classified as cathodic or anodic if the difference in corrosion potential is more than 85 mV with respect to the corrosion potential of the blank [29, 30]. Such results indicate that the CFF extract acts as a mixed-type inhibitor with predominant cathodic effectiveness. The *LPR* value increases from 501 to 1335 Ohm·cm^2. The corrosion current decreases from $7.688 \cdot 10^{-5}$ to $2.593 \cdot 10^{-5}$ A/cm^2. These observations confirm that a protective film is formed on the metal surface. This controls the corrosion of metal [31–35]. The inhibition efficiency calculated from polarization study comes to 62.47%. The difference in inhibition efficiencies between the weight loss method and polarization method may be attributed to the fact, that weight loss method is an average method after immersion period of one day, whereas polarization method is an instantaneous method.

Table 4. Corrosion parameters of MS immersed in SOWW in the absence and presence of inhibitor (CFF) obtained by polarization study.

System	E_{corr} mV *vs* SCE	b_c mV/decade	b_a mV/decade	*LPR* Ohm·cm²	I_{corr} A/cm²	*IE* %
SOWW	–831	150	216	501	$7.688 \cdot 10^{-5}$	–
SOWW+10% CFF	–875	132	202	1335	$2.593 \cdot 10^{-5}$	62.47

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 *Cassia fistula* flower were dried on a glass plate. A solid mass was obtained. It was blended with KBr and converted into pellet. Its FTIR spectral pattern has been recorded and given in Figure 4, the assignments are shown in Table 5. A strong broad band at 3426.34 cm^{-1} is assigned to –OH stretching group. The weak band observed at 2934.93 cm^{-1} is consistent with C–H stretching vibration. The sharp band at 1629.98 cm^{-1} due to C=O group. The peak at 1407.92 cm^{-1} indicates C–H bending vibrational frequency. The peak at 1073.88 cm^{-1} reveals the C–O–C stretching vibration.

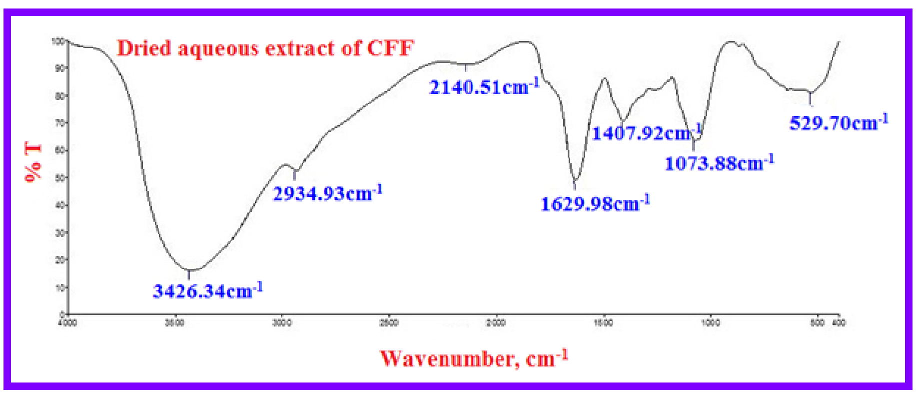


Figure 4. FTIR spectrum of dried aqueous extract of pure inhibitor (CFF).

The FTIR spectrum of the protective film formed on the mild steel surface immersion in the solution containing SOWW with 10% *v/v* of inhibitor (CFF) solution is shown in Figure 5. The respective absorption frequencies are given in Table 5. The slight shift at 3426.34 cm^{-1} to 3424.62 cm^{-1} can be attributed to the presence of –OH stretching. The peak shift at 2934.93 cm^{-1} to 2925.73 cm^{-1} is due to presence of C–H bond. The peak shifts from 1629.98 cm^{-1} to 1631.96 cm^{-1} indicate the presence of C=O group. The C–H bending frequency has shifted from 1407.92 cm^{-1} to 1384.20 cm^{-1}. The larger shifts from

1073.88 cm^{-1} to 1022.18 cm^{-1} is due to the presence of C–O–C stretching group. The new absorption band at 694.03 cm^{-1} and 473.44 cm^{-1} probably originates from the Fe^{2+} – *Cassia fistula* flower complex formation. This shows that due to the interaction between the metal and the active constituents, there is a change in the chemical nature of the active constituents [36, 37].

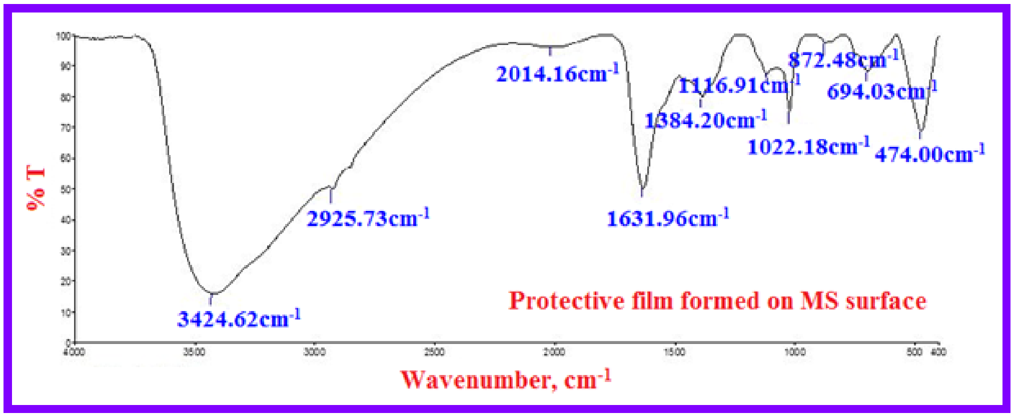


Figure 5. FTIR spectrum of scratched film from the mild steel surface after immersion in SOWW with 10% *v/v* aqueous extract of inhibitor (CFF).

Table 5. FTIR spectral data for the aqueous extract of CFF and the scratched film from mild steel surface after immersion in SOWW with 10% *v/v* CFF.

Stretching frequency, cm^{-1}		**Various functional groups**
Dried aqueous extract of CFF	**Protective film formed on mild steel surface**	
3426.34	3424.62	–OH stretching
2934.93	2925.73	–CH stretching
1629.98	1631.96	C=O stretching
1407.92	1384.20	C–H bending
1073.88	1022.18	C–O–C stretching
–	694.03	γ-Fe_2O_3
–	474.00	γ-Fe_2O_3

Analysis of SEM

SEM analysis has been widely used in corrosion inhibition study. SEM images have been recorded for polished mild steel surface, mild steel surface immersed in corrosive medium (SOWW) and mild steel surface immersed in corrosive medium containing the inhibitor (CFF) system. In the present study, SEM images were recorded for polished mild steel surface (System A), polished mild steel surface immersed in corrosive medium (SOWW) (system B) and polished mild steel surface immersed in corrosive medium (SOWW) containing the inhibitor (CFF) system (system C) [38, 39]. The images are shown in Figure 6(a, b, c). It is observed that for system A (Figure 6a) the surface is very smooth. For system B (Figure 6b) the surface is very rough. Pits are noticed due to corrosion. For system C (Figure 6c) the surface is smooth, due to the formation of protective film.

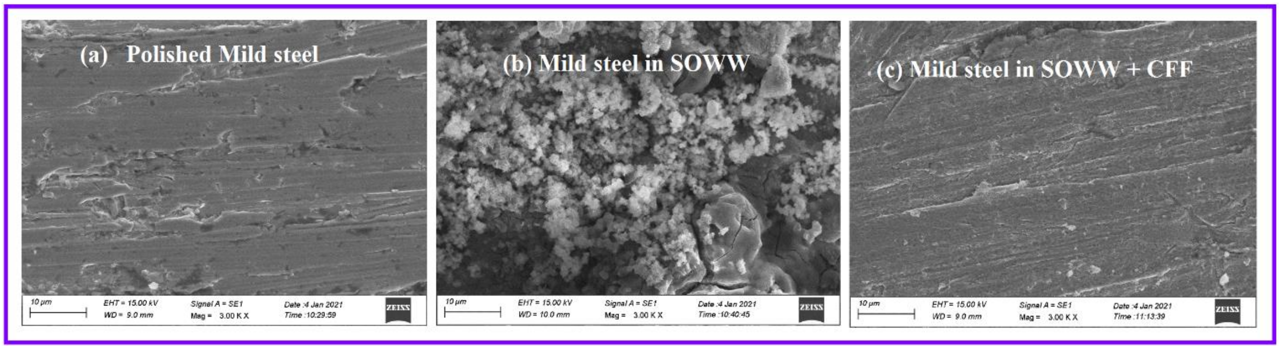


Figure 6. SEM images of (a) Polished mild steel (b) mild steel immersed in SOWW (c) mild steel immersed in SOWW containing inhibitor (CFF).

Analysis of AFM

Atomic force microscopy is used to analyze the surface morphology of the testing specimen [40, 41]. The two-dimensional and the three-dimensional AFM images of polished MS surface, MS immersed in SOWW and also in inhibitor (CFF) system are shown in Figures 7 and 8. Roughness parameters obtained from AFM studies such as Root Mean Square (R_q) roughness (nm), Average (R_a) roughness (nm) and maximum peak-to-valley height (nm) are summarized in Table 6. It is observed from the Table 6 that the AFM parameters associated with roughness such as the root mean square roughness, average roughness and maximum peak-to-valley height of polished MS surface are very low. The values are significantly very high for MS surface in SOWW (corrosive medium). In the case of MS in SOWW medium in presence of inhibitor (CFF) the values are somewhat higher than that of polished MS surface and considerably lower than that of the MS in SOWW (corrosive medium). These results reveal that a thin protective film is formed on the MS surface and prevents the MS from corrosion.

Table 6. AFM parameters of mild steel surface in the presence and absence of inhibitor (CFF) 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 CFF extract	185.59	157.85	683.37

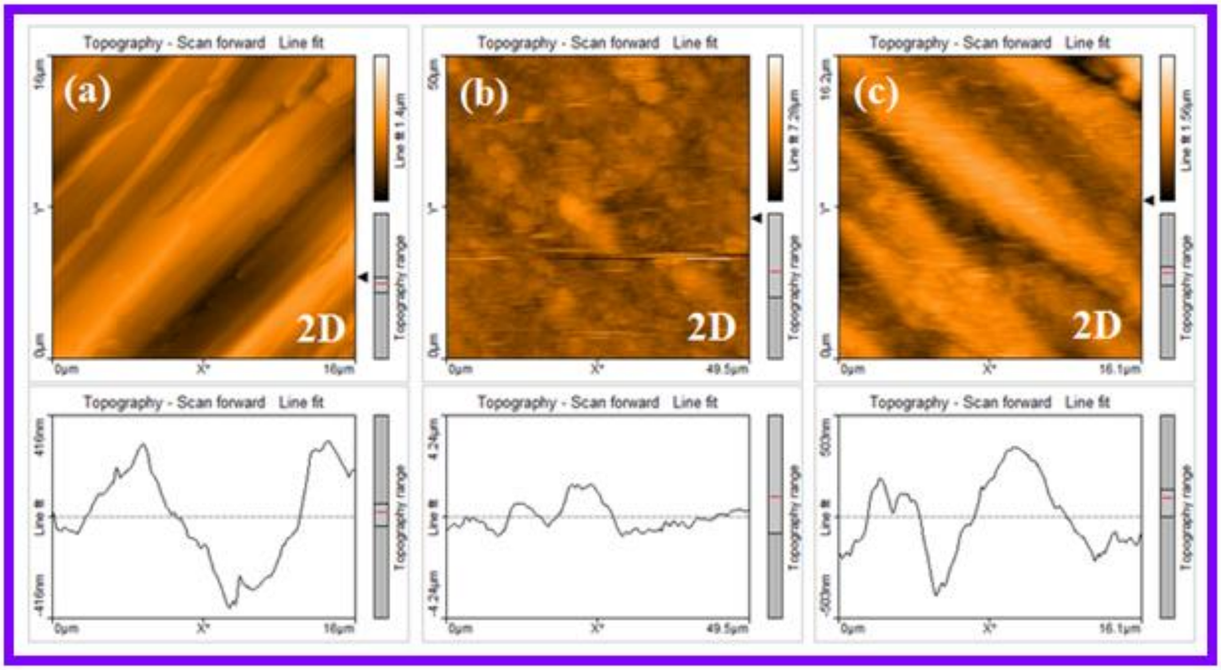


Figure 7. Two dimensional AFM images (a) polished MS, (b) MS immersed in SOWW and (c) MS immersed in SOWW containing inhibitor (CFF) system.

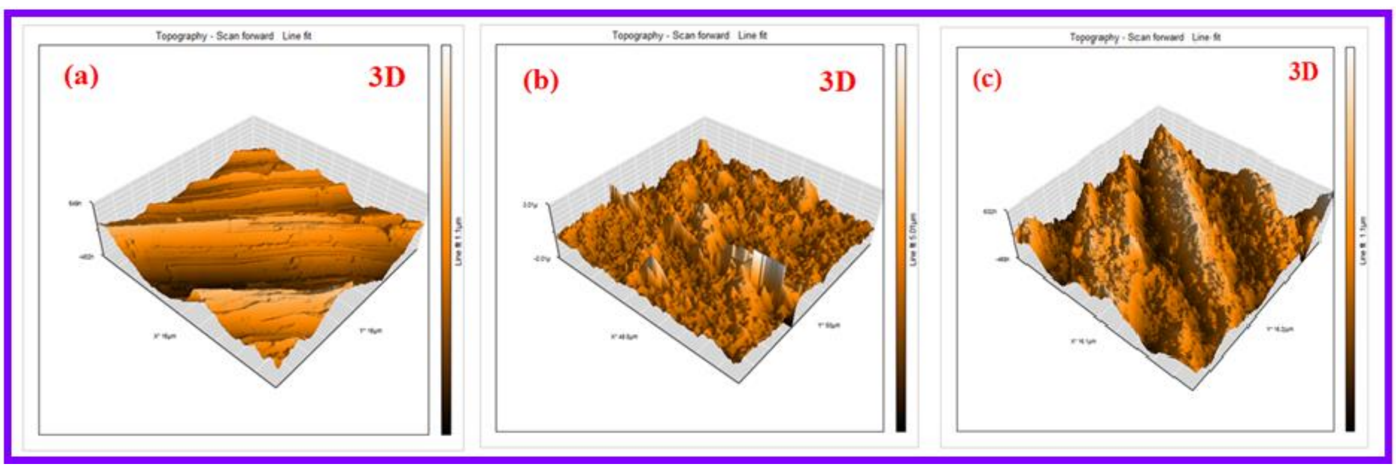


Figure 8. Three dimensional AFM images of the surface (a) Polished MS, (b) MS immersed in SOWW and (c) MS immersed in SOWW containing inhibitor (CFF) system.

Conclusion

The present study leads to the following conclusions.

- The GC-MS study suggests that *Cassia fistula* flower **(**CFF) has anti-corrosion property on mild steel owing to the presence of hetero atoms present in the active principles of the extracts of the natural product.
- FTIR results have proved the presence of functional groups containing hetero atoms which are responsible for the formation of protective layer on metal surface interface.

- CFF shows maximum inhibition efficiency at 10% *v/v* of extract concentration as revealed by weight loss method.
- Polarization study reveals that this formulation functions as a mixed type of inhibitor system.
- The SEM and AFM analysis have shown that the inhibition of mild steel occurred by the development of inhibitive film on the metal surface.
- The CFF extract function as an efficient inhibitor in controlling corrosion of mild steel pipeline carrying SOWW.

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