Asafoetida Extract (ASF) as green Corrosion Inhibitor for Mild Steel in Sea Water

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Abstract

An aqueous extract of asafoetida has been used as a corrosion inhibitor in controlling corrosion of carbon steel. The main constituent of this extract is umbelliferone. It has excellent inhibition efficiency (IE) of 98% at Zn^{2+} (25 ppm) by the weight loss method. The protective film has been analyzed using Atomic Force Microscopic (AFM) and FTIR spectroscopic techniques. Protective film formed on the metal surface is confirmed by using Electro chemical studies such as potentiodynamic polarization technique. Polarization study reveals that this system functions as mixed type of inhibitor.

Key words: Carbon steel, inhibition efficiency, umbelliferone, AFM; green inhibitors, protective film.

Introduction

So many natural products are used for analyzing antimicrobial[1,2,3], antifungal[4], anticancer[2], antibacterial activity[5] in several areas. Nowadays need for growing corrosion inhibitors becomes increasingly necessary to stop or delay the attack of a metal in an aggressive solution. Considerable efforts are made to find suitable compounds to be used as corrosion inhibitors in various corrosive media. Many works were conducted to examine extracts from naturally substances. So in this work we have taken natural product as our corrosion inhibitor. Some references are coated here in which natural products are used as corrosion inhibitor. Corrosion inhibition of carbon steel in low chloride media by an aqueous extract of Hibiscus rosa - sinensis Linn has been evaluated by mass – loss method and electrochemical studies[6], corrosion inhibition by beet root extract in well water[7], electrochemical studies confirm the formation of a protective film on the metal surface by spirulina solution, this offers 90% corrosion inhibition efficiency[8] corrosion resistance of metals inartificial saliva in the absence and also in the presence of spirulina[9], corrosion behavior of aluminium in various media has been used to control corrosion of aluminium. To prevent the corrosion of aluminium in acid medium, inhibitors such as Chlomolaena Odorata L.[10], Ananas Sativum[11], Ipomoea Invulcrata[12].

There are several reviews on the use of plant extracts as corrosion inhibitors[13].Recently aqueous extract of Cocos nucifera - Coconut Palm – Petiole[14]. Fennel (Foeniculum Vulgare) Essential[15]. Pericarp of the Fruit of Garcinia Mangostana[16]. Natural[17]. Ethanol extract of Vernonia Amygdalina[18] and Ipomoea involcrata[19] have been used as corrosion inhibitors. Langmuir adsorption isotherm proved the effects of Alovera on corrosion of Zinc in HCl solution[20], in the presence of fruit peel in hydrochloric acid on carbon steel[21], and it also proved that Murraya Koenigii acts as corrosion inhibitor on mild steel in hydrochloric acid and sulphuric acid solutions[22], investigation of natural inhibitors is particularly interesting because they are non –expensive, ecologically friendly acceptable and possess no threat to the environment. Asafoetida is an ingredient of a plant mixture reported to have antidiabetic properties in rats[23], Asafoetida has a broad range of uses in traditional medicine as an antimicrobial, antiepileptic, used for treating chronic bronchitis and whooping cough[24,25]. The present work is undertaken: i. To evaluate the inhibition efficiency (IE) of an aqueous extract of asafotida (ASF) in controlling the corrosion of carbon steel in sea water, in the absence and presence of Zn^{2+} ii. To investigate the influence of immersion period on the IE of the system. iii. To analyze the protective film formed on the carbon steel by FTIR spectra, Polarization study and Atomic Force Microscope techniques.

Material and Methods

Preparation of the specimen: Carbon steel specimens of size 1.0 cm × 4.0 cm × 0.2 cm and chemical composition 0.026 % sulphur, 0.06 % phosphorous, 0.4 % manganese, 0.1 % carbon and the rest iron were polished to a mirror finish and degreased with trichloroethylene and used for the weight loss method and surface examination studies.

Preparation of umbelliferone asafoetida extract: An aqueous extract of umbelliferone asafoetida was prepared by grinding 5g of asafoetida, with distilled water, filtering the suspending impurities, and making up to 100 ml. The extract was used as corrosion inhibitor in the present study.

Weight-loss method: Carbon steel specimens were immersed in 100 ml of the sea water in [Nagarkovil, Tamil nadu, India] containing various concentrations of the inhibitor asafotida (ASF) in the absence and presence of Zn^{2+} for one day. The weights of the specimens before and after immersion were determined using a Digital Balance (Model AUY 220 SHIMADZU). The corrosion inhibition efficiency (IE) was then calculated using the equation
$IE = 100 [1-(W_2/W_1)]$ %

Where W_1 is the weight loss value in the absence of inhibitor and W_2 is the weight loss value in the presence of inhibitor.

Surface examination study: The carbon steel specimens were immersed in various test solutions for a period of 1 day. After 1 day, the specimens were taken out and dried. The nature of the film formed on the surface of the metal specimen was analyzed by various surface analysis techniques.

Fourier transform infrared spectra: These spectra were recorded in a Perkin-Elmer-1600 spectrophotometer using KBr pellet. The FTIR spectrum of the protective film was recorded by carefully removing the film, mixing it with KBr and making the pellet.

Atomic Force Microscopy characterization (AFM): The carbon steel specimen immersed in blank and in the inhibitor solution for a period of one day was removed, rinsed with double distilled water, dried and subjected to the surface examination. Atomic force microscopy (Veeco dinnova model) was used to observe the samples' surface in tapping mode, using cantilever with linear tips. The scanning area in the images was 5 µm × 5 µm and the scan rate was 0.6 HZ /second.

Potentiodynamic Polarization: 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. The working electrode was carbon steel. A SCE was the reference electrode. Platinum was the counter electrode. From polarisation study, corrosion parameters such as corrosion potential (E_{corr}), corrosion current (I_{corr}), Tafel slopes anodic = b_a and cathodic = b_c were calculated and polarization study was done. The scan rate (V/S) was 0.01. Hold time at (Efcs) was zero and quiet time (s) was two.

Table-1
Parameters of Sea water [Nagarkovil, Tamil nadu, India]

parameters	Value
pH	7.4
Electrical conductivity	58564 micS/cm
Total disolved soilds	39824 ppm
Total Hardness	112 ppm
Magnesium	14 ppm
Calcium	21 ppm
Chloride	18350 ppm
Sulphate	4354 ppm

Analysis of results of weight - loss study: The calculated Inhibition efficiencies (IE) and corresponding corrosion rates of asafoetida (ASF) in controlling the corrosion of carbon steel immersed in the presence and absence of Zn^{2+} have been tabulated in table-2

It is observed that asafoetida only shows good inhibition efficiency (IE) (in the absence of Zn^{2+}). When Zn^{2+} (5 ppm) is added IE also increases .IE increases and gives maximum 98 % IE at 4 ml of ASF and 25 ppm of Zn^{2+} this shows that synergistic effect exists between Zn^{2+} and the active principles present in ASF. When the concentration of Zn^{2+} increases from 5 ppm to 25 ppm the IE slightly decreases. This may be due to the fact that, when the concentration of Zn^{2+} increases, the Zn^{2+}-ASF complex formed is precipitated in the bulk of the solution. Hence ASF is not transported towards the metal surface. So the IE decreases,similar observation was made with Hibiscus Rosa-Sinensis Al at pH 12[26] Euphorbia[27] Henna[28].

Table-2
Corrosion rates (CR) and inhibition efficiency of carbon steel immersed in an aqueous solution in The absence and presence of inhibitors

ASF (ml)	IE%				CR mm/y			
	Zn^{2+} (ppm)				Zn^{2+} (ppm)			
	0	5	15	25	0	5	15	25
0	--	20	25	30	0.1623	0.1298	0.1217	0.1136
2	60	54	54	88	0.0649	0.0746	0.0746	0.0194
4	65	62	60	98	0.0568	0.0616	0.0649	0.0032
6	71	65	65	94	0.0470	0.0568	0.0568	0.0097
8	71	77	82	88	0.0470	0.0373	0.0292	0.0194

Inhibitors : asafoetida (ASF) + Zn^{2+} Period of immersion : 1 day

Table-3
Influence of duration of immersion on the inhibition efficiency of ASF-Zn^{2+} system

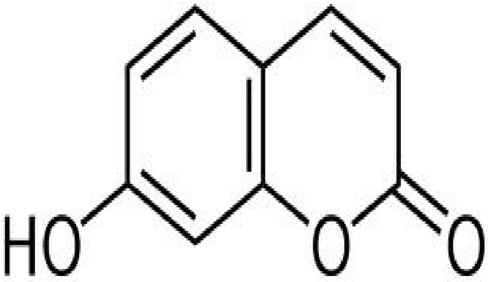
Immersion period, day	1	3	5	7
CR in the absence of the Inhibitor, mdd	0.1623	0.3153	0.4545	0.6214
CR in the presence of the inhibitor, ASF (4ml) +Zn^{2+} (25 ppm)	0.0032	0.1387	0.2999	0.4598
IE,%	98	56	34	26

Influence of immersion period on the inhibition efficiency of ASF: The influence of duration of immersion on the IE of ASF (4ml) – Zn^{2+} (25ppm) system is given in table-3.When the immersion period increases the inhibition efficiency decreases and the corrosion rate increases. This shows that the protective film formed on the metal surface, was broken by the corrosive environment and the film was dissolved, this same result is shown in Phyllanthus amarus extract[29], it is observed that in phyllanthus amarus as the duration of immersion increases the IE decreases.

Results and Discussion

The physicochemical parameters of Sea water are given in table-1.

Analysis of FTIR spectra: Earlier researchers have confirmed that FTIR spectrometer is a powerful instrument that can be used to determine the type of bonding for organic inhibitors adsorbed on the metal surface[30]. FTIR spectra have been used to analyze the protective film formed on metal surface. FTIR spectrum of pure dried Asofoetida ASF is given in figure-1a. The FTIR spectrum of the film formed on the metal surface after immersion in the sea water for 1 day containing 4ml of ASF and 25 ppm of Zn^{2+} is shown in Fig-1b. The OH stretching frequency has increased from3407 cm^{-1} to 3425 cm^{-1},C=O stretching frequency has shifted from 1630 cm^{-1} to 1634cm^{-1}, ring O stretching frequency has increased from 1039 cm^{-1} to 1056 cm^{-1} [31]. The band due to conjugated double bonds shifts from 3785 cm^{-1} to 3751 cm^{-1}.This indicates that the oxygen atom of C=O group has coordinated with Fe^{2+} formed on the metal surface resulting in the formation of Fe^{2+}- ASF complex on the metal surface. The peak at 1376 cm^{-1} is due to $Zn(OH)_2$ formed on the cathodic sites of the metal surface[29]. The active principle in an aqueous extract of asafotida is (umbelliferone) this figure is shown in scheme:1.



umbelliferone
Scheme :1

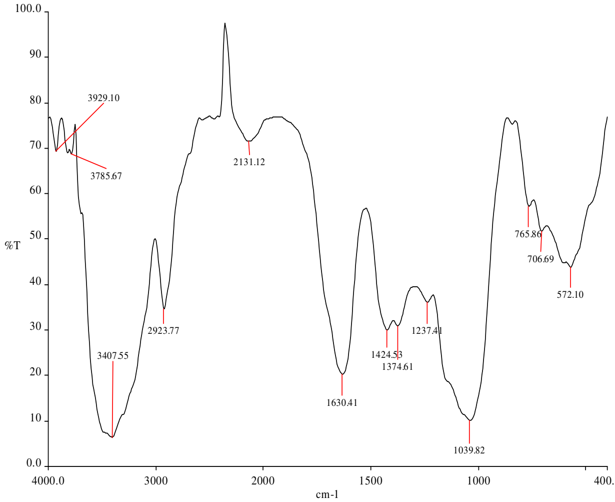


Figure-1a
FTIR spectrum of pure dried ASF

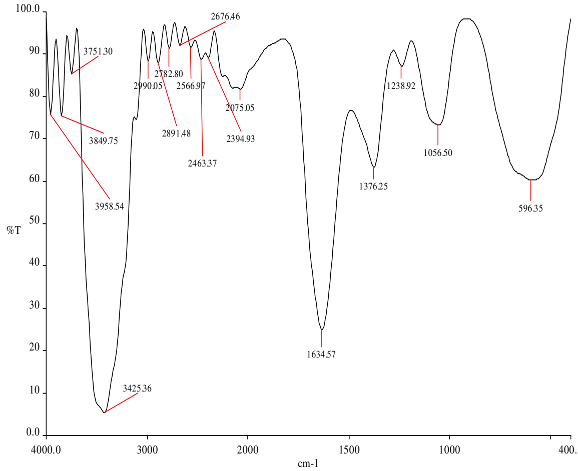
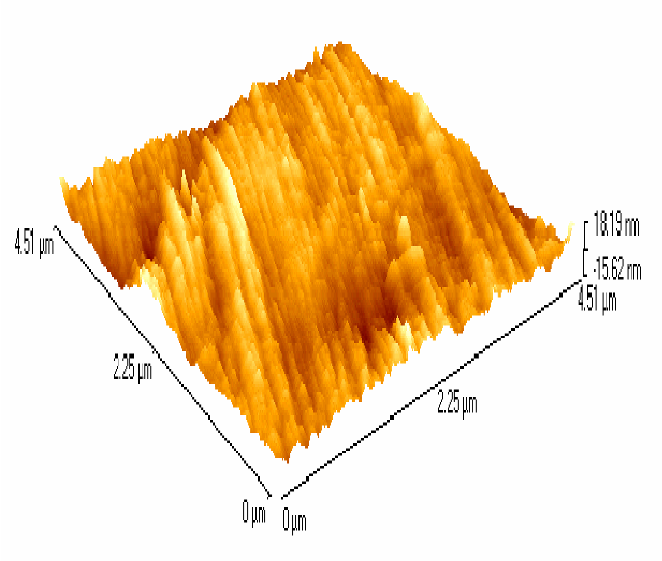
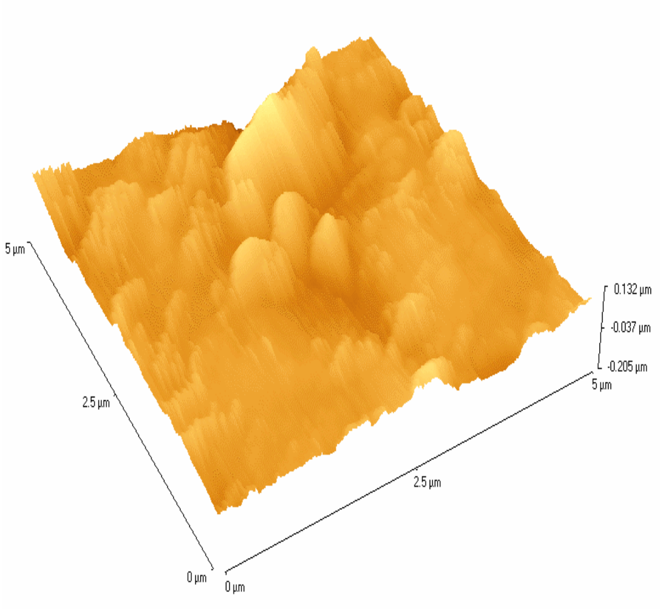


Figure-1b
FTIR spectrum of the film formed on the metal surface after immersed in the sea water containing 4ml of ASF and 25 ppm of Zn^{2+}

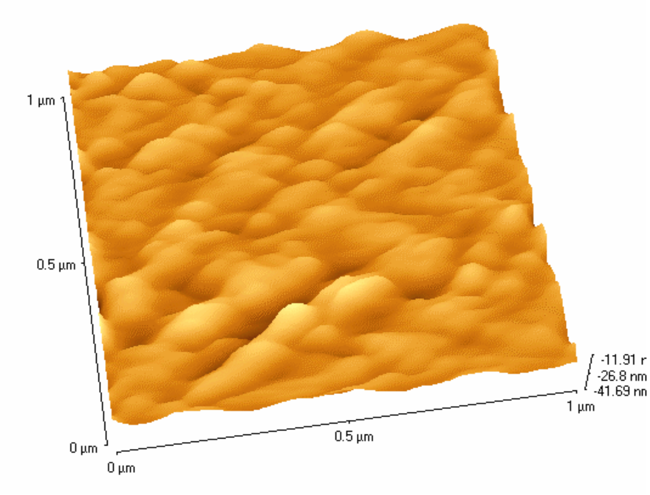
Atomic Force Microscopy Characterization: AFM is a powerful technique to investigative the surface morphology at nano- to micro-scale and has become a new choice to study the influence of inhibitor on the generation and the progress of the corrosion at the metal/solution interface[32-35]. The three dimensional (3D) AFM morphologies and the AFM cross-sectional profile for polished carbon steel surface (reference sample), carbon steel surface immersed in sea water (blank sample) and carbon steel surface immersed in sea water containing the formulation of 4ml of BPE and 15 ppm of Zn^{2+} are shown as figure-2 images (a, d,), (b, e,), (c, f,) respectively. Root– mean-square roughness, average roughness and peak-to-valley value AFM image analysis was performed to obtain the average roughness, Ra (the average deviation of all points roughness profile from a mean line over the evaluation length), root-mean-square roughness, Rq (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)[33].



(a)

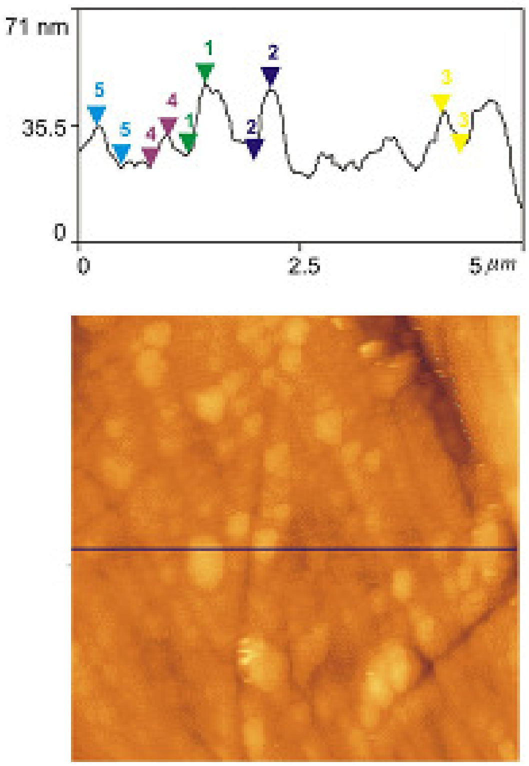


(b)

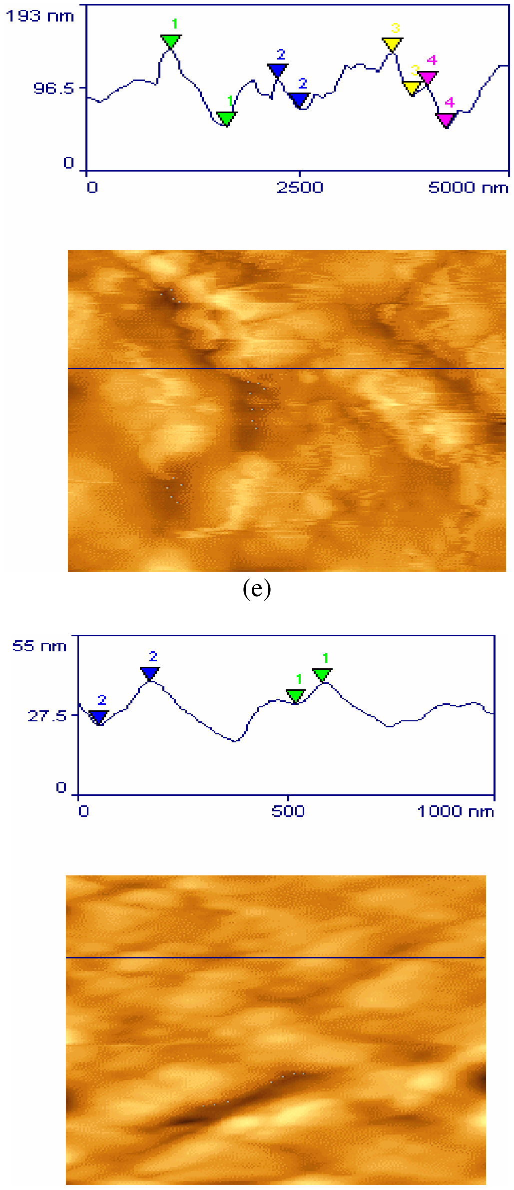


(c)

Figure-2
Three dimensional AFM images of the surface of: a) As polished carbon steel(control); b) carbon steel immersed in sea water (blank); c) carbon steel immersed in sea water containing ASF (4ml) + Zn^{2+} (25ppm)



(d)



(e)

(f)

Figure-2

AFM cross-sectional images of the surface of: d) As polished carbon steel (control); e) carbon steel immersed in sea water (blank); f) carbon steel immersed in sea water containing ASF (4ml) + Zn^{2+} (25ppm)

Table-4 is a summary of (Rq), (Ra), (P-V) value for carbon steel surface immersed indifferent environment. Figure-2 (a, d,) displays the surface topography of un-corroded metal surface. The value of Rq, Ra and P-V height for the polished carbon steel surface (reference sample) are 4.3 nm, 3.41nm and 35.28 nm respectively. The slight roughness observed on the polished carbon steel surface is due to atmospheric corrosion. Figure- 2 (b, e,) displays the corroded metal surface with few pits in the absence of the inhibitor immersed in sea water. The (Rq), (Ra), (P-V) height values for the carbon steel surface are 25.2nm, 19.9nm and 89.10 nm respectively. These data suggests that carbon steel surface immersed in sea water has a greater surface roughness than the polished metal surface, which shows that the unprotected carbon steel surface is rougher and was due to the corrosion of the carbon steel in sea water environment. Figure-2 (c, f,) displays the steel surface after immersion in sea water containing 4ml of ASF and 25 ppm of Zn^{2+} . The (Rq), (Ra), (P-V) height values for the carbon steel surface are 6.38nm, 4.72nm and 14.81nm respectively The (Rq), (Ra), (P-V) height values are considerably less in the inhibited environment compared to the uninhibited environment. These parameters confirm that the surface is smoother. The smoothness of the surface is due to the formation of a compact protective film of Fe^{2+} - ASF complex thereby inhibiting the corrosion of carbon steel[33].

Analysis of polarization curve: The potentiodynamic polarization curves of carbon steel immersed in sea water in the absence and presence of inhibitors (Asafotida extract (ASF) and Zn^{2+}) are shown in figure-3. The corrosion parameters namely corrosion potential (E_{corr}), Tafel slopes (b_c = cathodic b_a = anodic), linear polarization resistance (LPR) and corrosion current (I_{corr}) are given in table-5. It is observed that in the absence of inhibitors the corrosion potential is-771 mV vs SCE. In the presence of inhibitors (4ml ASF and 25 ppm Zn^{2+}) the corrosion potential is shifted to -765 mV vs SCE .The shift is very small this suggests that this formulation controls the anodic reaction and cathodic reaction to an equal extant . So we can conclude that this inhibitor acts as mixed type of inhibitor .The LPR value increases from 2378 ohm cm^2 to 5664 ohm cm^2 further the corrosion current decreases from 1.579 $x10^{-5}$ A/cm^2 to $0.6832x10^{-5}$ A/cm^2 .This suggests that a protective film is formed on the metal surface[36-40].

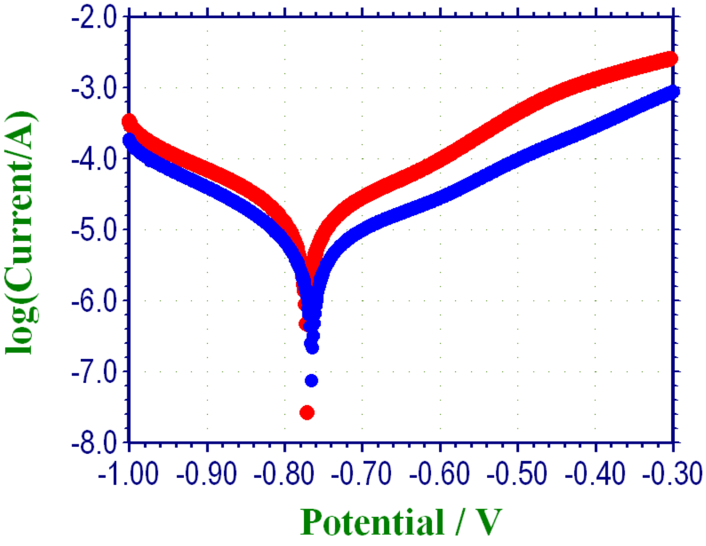


Figure-3

Polarization curves of carbon steel immersed in various test solutions (a) sea water (blank); (b) ASF (4ml) + Zn^{2+} 25 ppm

Table-4
AFM data for carbon steel surface immersed in inhibited and uninhibited environment.

Samples	RMS(Rq) Roughness (nm)	Average(Ra Roughness (nm)	Maximum Peak-to valley Height (nm)
1.Polished carbon steel	4.33	3.41	35.28
2.Carbon steel immersed in sea water (blank)	25.2	19.9	89.10
3.Carbon steel immersed in sea water + ASF (4ml) + Zn^{2+} (25ppm)	6.38	4.72	14.81

Table5
Corrosion parameters of carbon steel immersed in Asafotida extract obtained from polarization study.

System	E_{corr} mV vs SCE	b_c mV/decade	b_a mV/decade	LPR ohm cm^2	I_{corr} A/cm^2
Sea water(blank)	-771	160	186	2378	$1.579x10^{-5}$
ASF(4ml)+ Zn^{2+}(25ppm)	-765	148	222	5664	$0.6832x10^{-5}$

Conclusion

The present study leads to the following conclusions. i. The formulation consisting of 4ml of ASF and 25 ppm of Zn^{2+} offers 98% inhibition efficiency to carbon steel immersed in sea water. ii. When immersion period increases corrosion rate also increases. iii. Polarization study reveals that this system formulation acts as a mixed type of inhibitor. iv. The FTIR spectra reveal that the protecting film consists of Fe^{2+}-Asafotida (active ingredient) complex. v. AFM studies confirm that the surface is smoother. The smoothness of the surface is due to the formation of a compact protective film of Fe^{2+} - ASF complex on the metal surface thereby inhibiting the corrosion of carbon steel.

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