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Comparative Evaluation of Hibiscus Cannabinus and Solanum Trilobatum Extracts as Inhibitors for C- Steel Corrosion protection in Acidic Medium

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Abstract

This study analyzed the efficacy of various inhibitors in safeguarding carbon steel against corrosion in hydrochloric acid. The compounds that were subjected to testing included Acalypha indica root extract (AIRE), Hibiscus cannabinus (HC), and Solanum trilobatum (ST) leaf extracts. The investigation focused on the inhibitory efficacy through the application of gravimetric and electrochemical impedance methods. GC-MS and FTIR were employed to investigate the features of the inhibitors and co-inhibitors. Tafel data indicated that extracts from both leaves display mixed-type behaviour in inhibiting corrosion. The examination of AC impedance spectra indicated the formation of a surface protective coating, a finding that was supported by investigations using SEM and AFM techniques. The results indicated a significant increase in AIRE's inhibitory efficacy when it was used in conjunction with HC and ST co-inhibitors. On comparison, the maximum inhibition efficiency of 93% was obtained for the mixture of ST leaf extract with AIRE at a concentration of 20 ppm. A suitable inhibition mechanism has been proposed based on the active phytochemical constituents present in the extracts. Evidence from GC–MS and FTIR analyses indicates that the presence of a higher number of heteroatoms (N and O) along with aromatic π-electrons in the ST leaf extract is likely responsible for the enhanced protection of carbon steel in the acidic medium.

Keywords: Acalypha indica, Corrosion, Hibiscus cannabinus, Inhibitory efficiency, Solanum trilobatum

INTRODUCTION

Equipment for the production of oil and fertilizer are among the many industrial uses of carbon steel [1]. One typical pre-treatment method is pickling, which involves removing scale and rust using acids [2]. Because of the potential for acid mist and metal substrate corrosion, inhibitor usage is essential throughout this process [3]. Using inhibitors is a common practice to prevent corrosion [4]. A non-toxic and eco-conscious alternative for corrosion inhibitors is plant extracts. Easy and inexpensive methods exist for obtaining these extracts [5, 6]. Corrosion inhibitors degrade. Use of certain inhibitors, such as chromates, has been banned because of toxicity and the environmental hazards they produce. Hence there is a strive to make use of environmental friendly, non-hazardous / less toxic of naturally occurring plant materials as corrosion inhibitors in controlling the corrosion of mild steel is immersed in acids. Disposal of organic inhibitors are very difficult and non-biodegradable. Plant extracts' biodegradability restricts long-term use. Plant leaf aqueous extracts

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are cheap, non-toxic, safe, and accessible. To mitigates corrosion; phytochemicals included in plant extracts contain heteroatoms (such as sulfur, nitrogen, and oxygen) and π-electrons,enabling their adherence to metal surfaces [7, 8].

Comparative analyses of the inhibitive properties of plant extracts in corrosive medium have been reported to prevent corrosion [9–12]. The protective effect of C-steel in a 0.5N HCl solution was improved by adding a little amount of co-inhibitors (HC and ST) to AIRE, according to this research. For this study, researchers in Dindigul gathered leaves of two plants: HC, also known as Deccan hemp or Kenaf, and ST, whose Tamil name is Thuthuvalai (meaning climbing brinjal or three lobed nightshade in English) [13]. Through the gravimetric approach, we determined that 50 ppm AIRE, in conjunction with HC and ST leaf extracts, mitigated C-Steel corrosion in an HCl solution. Atomic force microscopy and scanning electron microscopy were used to assess the effectiveness of the inhibitors and co-inhibitors on the metal surface.

MATERIALS AND METHODS

Preparation of Material

This analysis used C-steel procured from the market. The composition consists of 0.27% carbon, 98.031% iron, and trace amounts of alloying metals like silicon, manganese, sulphur, phosphorus, nickel, and copper. The specimens were prepped for future use by polishing them using silicon carbide waterproof paper until evenly covered. Subsequently, they were cleaned using acetone.

Preparation of Extract

Following the collection of the fresh AIRE and HC roots and leaves from a nearby garden, we proceeded to dry them in the shade for duration of four to five days. The desiccated leaves were processed into a fine powder and subsequently stored in an airtight container to prevent air exposure. The ethanolic extract was prepared by refluxing 100 milliliters of ethanol with ten grams of powdered roots and leaves in a Soxhlet device for duration of three hours. Following the cooling process, the solution underwent filtration and was subsequently allowed to evaporate. In each sample, one milliliter of double-distilled water was used to dissolve one gram of residue. This extract has been used in subsequent research.

Bioactive Compound Analysis

The AOC-20i, Shimadzu system conducted GC-MS analysis on AIRE, HC, and ST extracts using a capillary column Elite-5 at a temperature of 280°C in split injection mode, with a flow rate of 1.20 ml/min. The acquired spectrum can facilitate the analysis of unidentified chemicals by allowing for a comparison of its components with those of known substances found in the NIST 2020 database.

Mass Loss Technique

A corrosive solution comprising 100 mL of 0.5N HCl, 50 ppm of AIRE, and varying concentrations of the co-inhibitor (range from 0 to 30 ppm) was used to suspend the polished and pre-weighed material samples. The test lasted for three hours at 303K. After three hours in the test solution, the specimens were taken out, cleaned with distilled water, dried, placed in a desiccator, and weighed. Equation may be used to calculate the IE percentage:

$$IE(\%) = \frac{Wb-Wi}{Wb} \text{X } 100 \tag{1}$$

The inhibited solution experiences a weight loss (Wi), whereas the corrodent solution (Blank) also undergoes a weight loss (W_b).

A mathematical approach was used to compute the corrosion rate, CR (miles per year):

$$CR(miles\ per\ year) = \frac{\text{534 xWeight Loss of specimen}}{\text{DAT}} \tag{2}$$

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A stands for the specimen's area in square inches, D for the density of carbon steel in grams per cubic centimeter, T for the length of immersion in hours, and W for the weight loss in milligrams [14].

Potentiodynamic Polarization Measurement

The polarization studies are conducted using a CHI electrochemical workstation, specifically model 608 E. The setup consists of three electrodes: a platinum counter electrode, a saturated calomel/carbon steel reference electrode, and a carbon steel coupon working electrode with a 1 cm^2 exposed surface area.

FT-IR Spectroscopy Studies

FTIR analysis of HC and ST leaf extracts was conducted using a SHIMADZU spectrophotometer through the KBr pellet method.

Surface Profiling Studies

Atomic Force Microscopy (AFM, PicoSPM 9500-21 with a scan rate of 6.835 µm/sec) and Scanning Electron Microscopy (SEM, particularly a JEOL model 6390 computer-assisted SEM) were used to investigate the surface changes caused by corrosion and the corrosion prevention procedures.

RESULTS AND DISCUSSION

GC-MS Analysis

The chemical structures of the bioactive substances found by GC-MS analysis of the inhibitor and co-inhibitors are shown in Figure 1. The presence of carbon double bonds and oxygen-containing functional groups, including hydroxyl and carbonyl, defines the characteristics of these molecules. Their capacity to adsorb onto carbon steel surfaces increases when they experience protonation in an acidic environment. The co-inhibitor ST, characterized by a greater number of heteroatom's, demonstrates a more effective capacity to inhibit corrosion on C-steel surface compared to the HC co-inhibitor, due to the presence of these functional groups [15].

Analysis of Weight Loss Data

The weight loss data for C-steel in a corrosive media is shown in Figure 2, which contrasts situations with and without an inhibitor at concentrations ranging from 50 to 300 ppm during various immersion times.

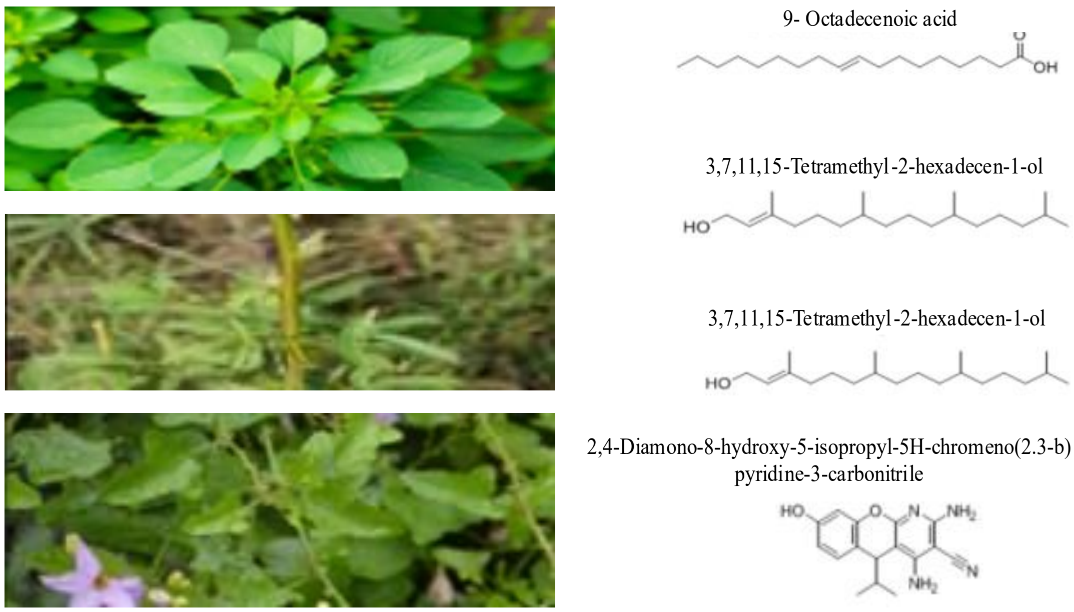

Figure 1. Chemical constituents present in the extracts (a) AIRE (b) HC (c) ST

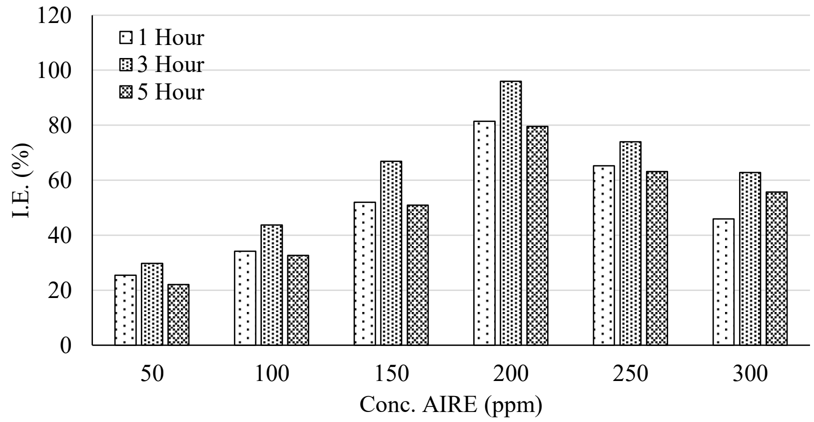

Figure 2. IE percentage of AIRE across different immersion durations.

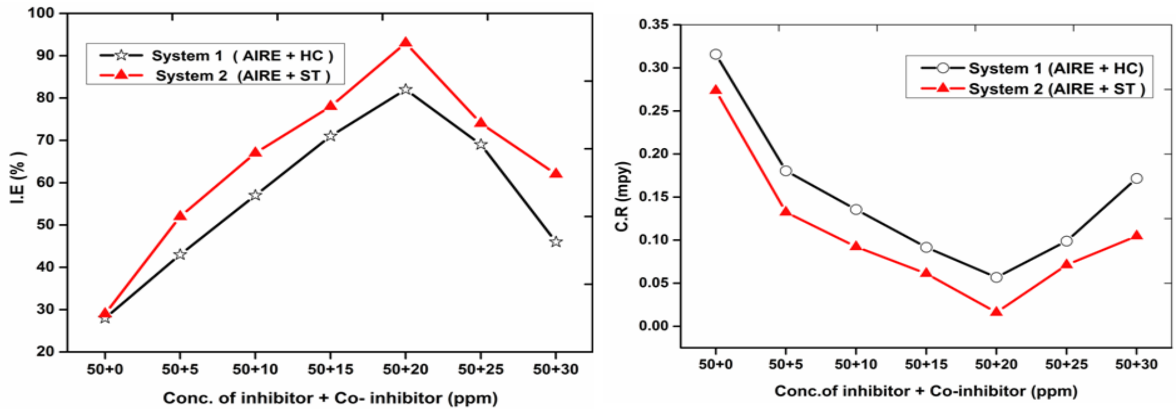

Figure 3. (a) IE (b) CR at various concentrations of system 1&2.

The graph shows that the inhibition effectiveness rose as the inhibitor concentration increased, reaching a high at 200 ppm before declining to 300 ppm. The highest protective effectiveness achieved after three hours of immersion was 96%. The effectiveness was reduced when the immersion period was extended to five hours, most likely as a consequence of the inhibitor molecules' desorption from the metal surface [16]. Consequently, a three-hour immersion period was established for further research. When compared to the performance of each inhibitor when taken alone, the combination of inhibitors shows a greater level of inhibitory efficiency. Consequently, various quantities of 5 ppm, 10 ppm, 15 ppm, 20 ppm, 25 ppm, and 30 ppm were combined with a lower quantity of AIRE (50 ppm). Extracts from HC (system 1) and ST (system 2) are used to prevent carbon steel from corroding in acidic situations.

Figure 3 shows the weight loss measurement data for various concentrations of System 1 and System 2 after 3 hours of immersion at room temperature. The incorporation of plant extract resulted in an enhanced inhibition efficiency (IE) and a reduction in the corrosion rate (CR).The optimum concentration for System 1 was 20 ppm, achieving an IE of 82%, while System 2 reached a maximum IE of 93% at the same concentration. According to the results, System 2 is more effective than System 1 in preventing carbon steel from corroding in an atmosphere with 0.5N HCl [17].

Potentiodynamic Polarization Measurement

Potentiodynamic polarization curves of carbon steel in 0.5N HCl are shown in Figure 4, which contrasts circumstances with and without the addition of inhibitors and co-inhibitors. Table 1 lists the Potentiodynamic polarization characteristics, which are contrasted with those of the two co-inhibitors that were used in this investigation.

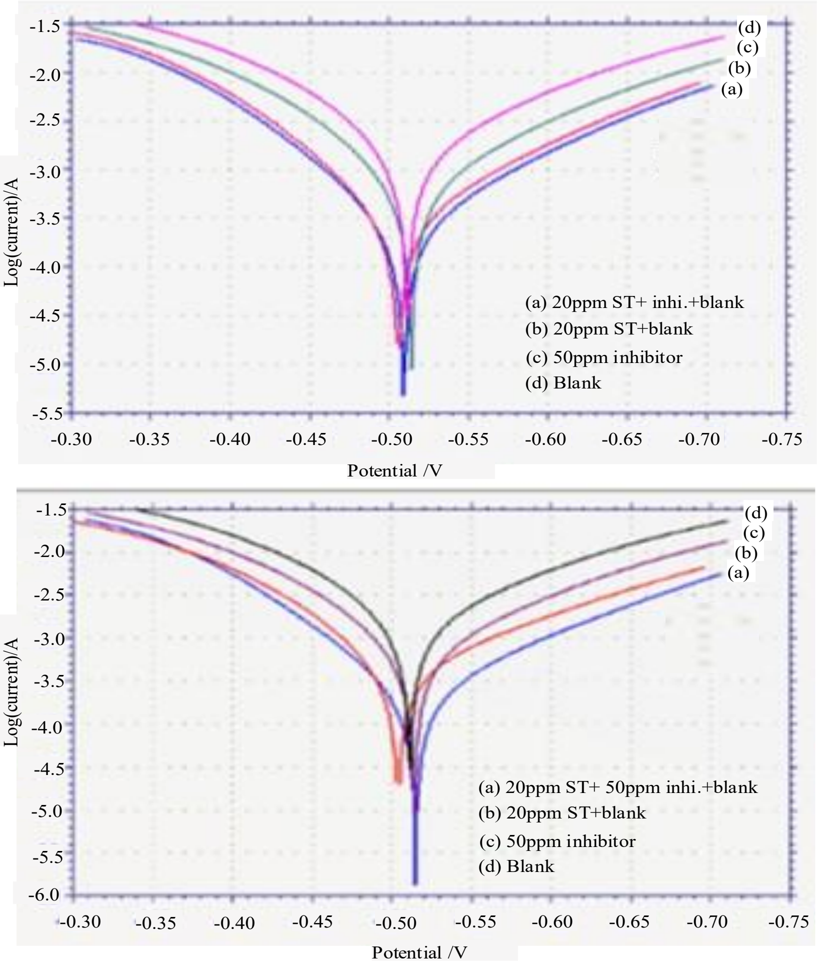

Figure 4. Tafel plots for C& S without and with mixed inhibitors in 0.5N Hcl solution.

Table 1. Polarisation measurement parameters.

System 1	**Conc. (ppm)**	**E_{corr} (V)**	**I_{corr} (A/cm$^{2)}$**	**β_a (V/dec)**	**β_c (V/dec)**	**LPR (Ohm /cm^{-2})**	**IE_i (%)**
	Blank	-0.512	$2.187x10^{-3}$	7.807	5.843	15.0	—
	50ppm inhi+ blank	-0.514	$9.662x10^{-4}$	8.796	5.926	30.6	56
	20ppm HC+ blank	-0.506	$5.226x10^{-4}$	10.231	6.054	43.9	76
	20ppm HC+ 50ppm inhi+ blank	-0.509	$3.775x10^{-4}$	11.048	6..979	63.9	82
System 2	Blank	-0.512	$2.187x10^{-3}$	7.807	5.843	15.0	—
	50ppm inhi+ blank	-0.514	$9.662x10^{-4}$	8.796	5.926	30.6	56
	20ppm ST+ blank	-0.504	$6.030x10^{-4}$	9.416	5.532	48.2	73
	20ppm ST + 50ppm inhi+ blank	-0.515	$2.479x10^{-4}$	12.008	7.563	89.6	89

Table 1 demonstrates that both System 1 and System 2 exhibit a decrease in corrosion current density concurrent with a rise in linear polarization resistance values. The significant changes noted in the anodic and cathodic polarization branches suggest that the inhibitors function as a mixed-type inhibitor. System 2 shows improved performance with maximum inhibition efficiency (IE) of 89%, attributed to its higher heteroatom content, compared to System 1 with an IE of 82% [18–20].

Analysis of AC Impedance Spectra

The investigation of film formation on the material surface was conducted using AC impedance spectra. The spectra presented in Figure 5 (a, b) along with the corresponding values are detailed in Table 2. This indicates that System 2 shows higher charge transfer resistance (R_{ct}) and lower double-layer capacitance (C_{dl}) values, suggesting the development of a protective film. This film formation is associated with high inhibition efficiency (IE) of 88% for System 2 [21, 23].

Table 2. AC impedance parameters of system 1 and 2.

System 1	**Conc. (ppm)**	**R_{ct} (Ohm-cm^2)**	**C_{dl} (F- cm^{-2})**	**IE_i (%)**
	Blank	13	6.96×10^{-7}	—
	50ppm inhi+ blank	35	2.58×10^{-7}	63
	20ppm HC+ blank	70	1.29×10^{-7}	81
	20ppm HC+ 50ppm inhi+ blank	86	1.05×10^{-7}	84
System 2	Blank	13	6.96×10^{-7}	—
	50ppm inhi+ blank	35	2.58×10^{-7}	63
	20ppm ST + blank	44	2.05×10^{-7}	71
	20ppm ST + 50ppm inhi+ blank	96	0.94×10^{-7}	88

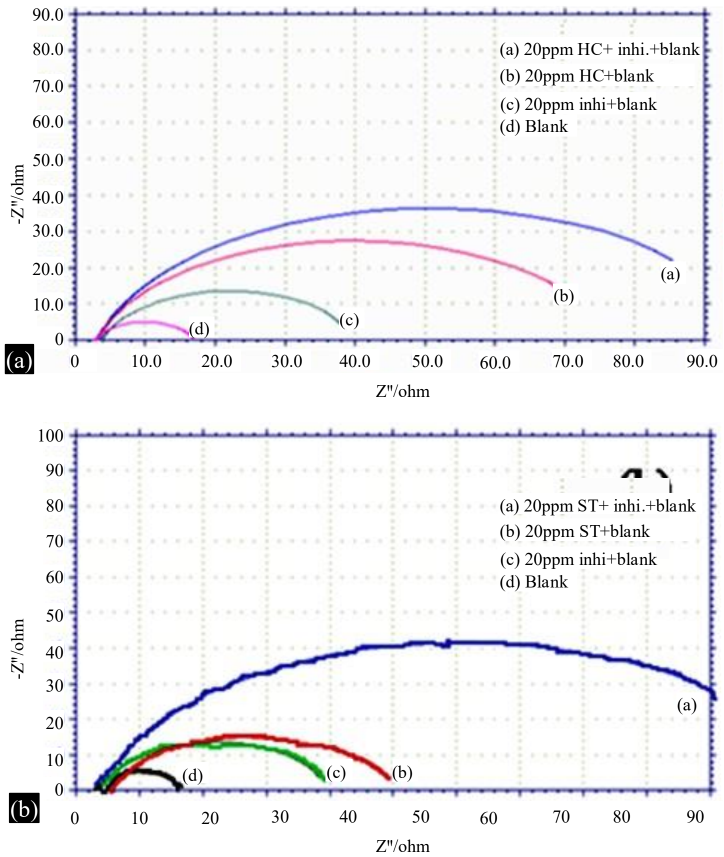

Figure 5. AC impedance spectra of various test solutions in (a) System1 (b) System 2.

FTIR Spectral Analysis

Both HC and ST leaf extract were subjected to a functional group analysis using the FTIR spectrum shown in Figure 6.

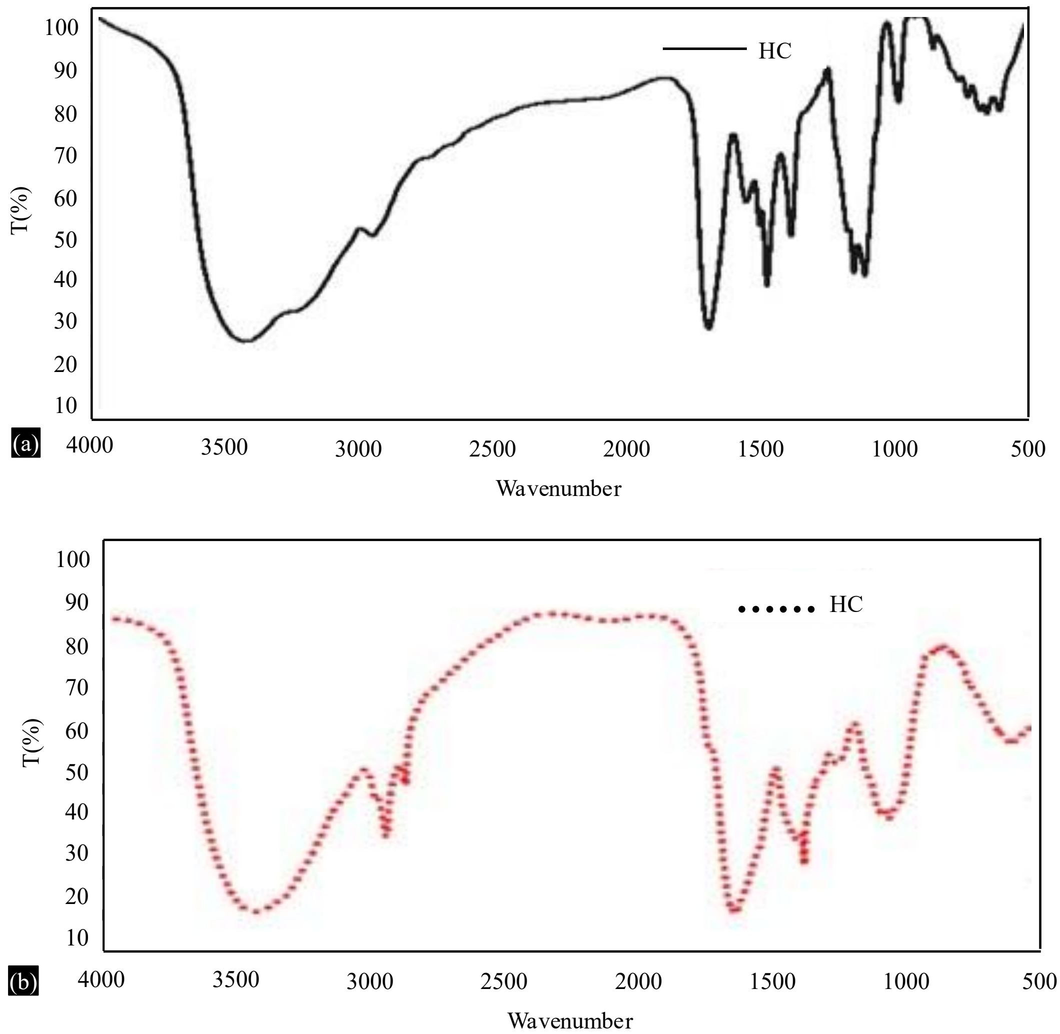

Figure 6. (a, b) FTIR spectrum of leave extracts of HC and ST.

The absorption bands associated with HC are attributed to the hydroxyl group (3428 cm^{-1}), alkyl C-H stretching (2929 cm^{-1}), C=C stretching (1634 cm^{-1}), C-O stretching (1075 cm^{-1}), and other C-H bending vibrations in the fingerprint region. Figure 6 above illustrates the absorption frequency range for ST and its constituent functional group. The primary amine exhibits O-H stretching / N-H stretching at 3408 cm^{-1} and C=C / C=N stretching at about 1632 cm^{-1}.

The spectra in the 2919 cm^{-1} and 2849 cm^{-1} region may be affected by the isopropyl group and other aliphatic bonds. The absorption peaks at 1386 cm^{-1}, 1267 cm^{-1}, and 1072 cm^{-1} are indicative of stretching vibrations associated with C-O, C-N, and C-C bonds, respectively. The hydroxyl or amine groups in the HC and ST extracts may promote the adhesion of organic molecules to the material's surface, hence reducing corrosion susceptibility [24, 25].

Surface Profiling Techniques

An SEM examination was conducted to examine the surface roughness of carbon steel under different situations. The results demonstrated that the polished carbon steel surface (Figure 7a) had a smooth texture and was devoid of corrosion products, whereas the surface exposed to 0.5N HCl (Figure 7b) revealed considerable corrosion damage. The incorporation of System 1 and System 2 inhibitors into the acidic solution (Figure 7c) led to the formation of a protective coating on the steel surface, which reduced corrosive damage [26, 27].

The AFM analysis, as exposed in Figure 8, reveals the surface topography of steel under different conditions. The polished surface had an average roughness (Ra) of 10.30 nm, which increased to 105.94 nm after exposure to acid. However, the addition of System 1 and System 2 inhibitors reduced the average roughness to 66.33 nm and 29.15 nm, respectively, indicating a significant protective effect [28].

Mechanism of Inhibition Action

Double or triple bonds, as well as heteroatoms like oxygen, nitrogen, sulphur, and phosphorus, are essential to the adsorption process. The GC-MS data indicates that the inhibitor's root extract has substantial organic constituents, including oxygen as a heteroatom (O–H, C–O), aligning with the characteristic features of organic corrosion inhibitors. 3, 7, 11, 15-Tetramethyl-2-hexadecen-1-ol serves

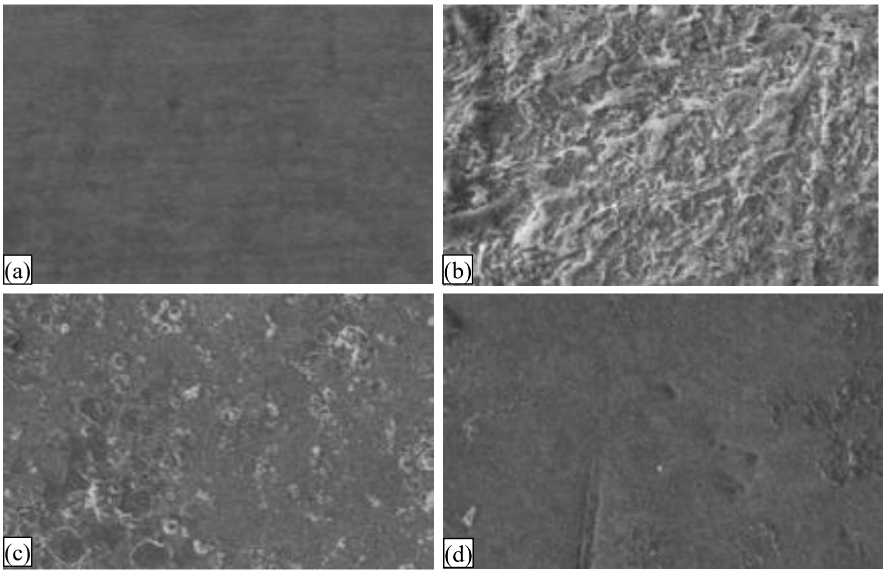

Figure 7. SEM representation for C&S (a) Control (b) Blank (c) System 1 (d) System 2.

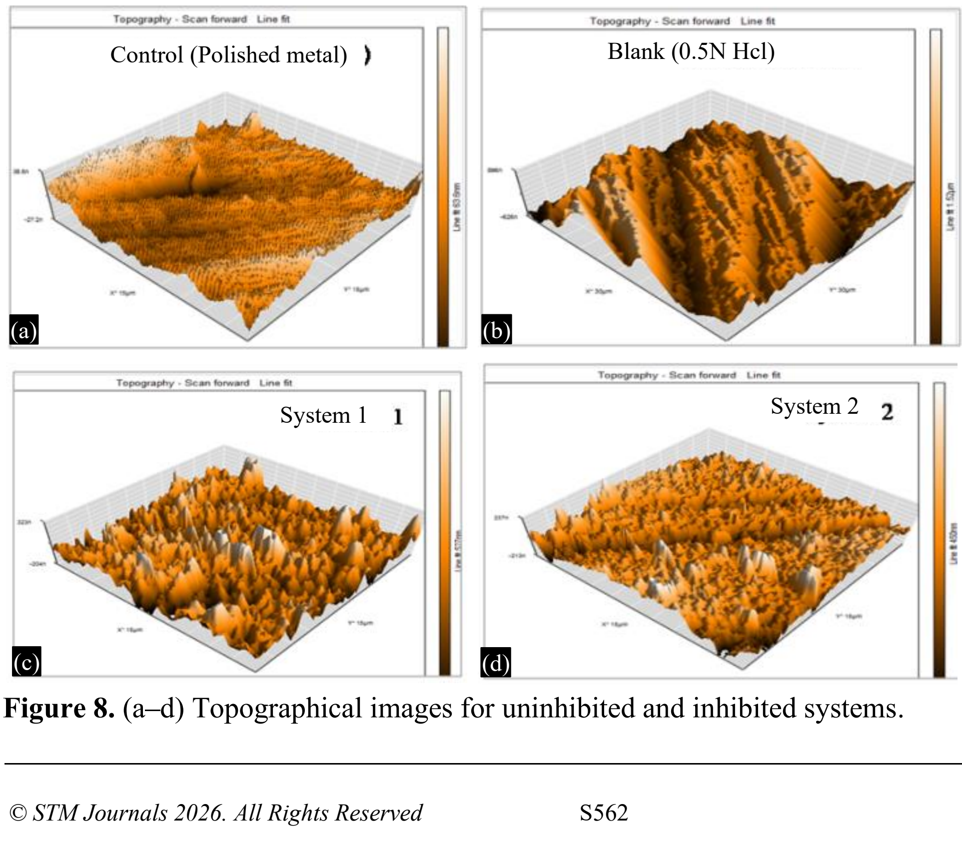

Figure 8. (a–d) Topographical images for uninhibited and inhibited systems.

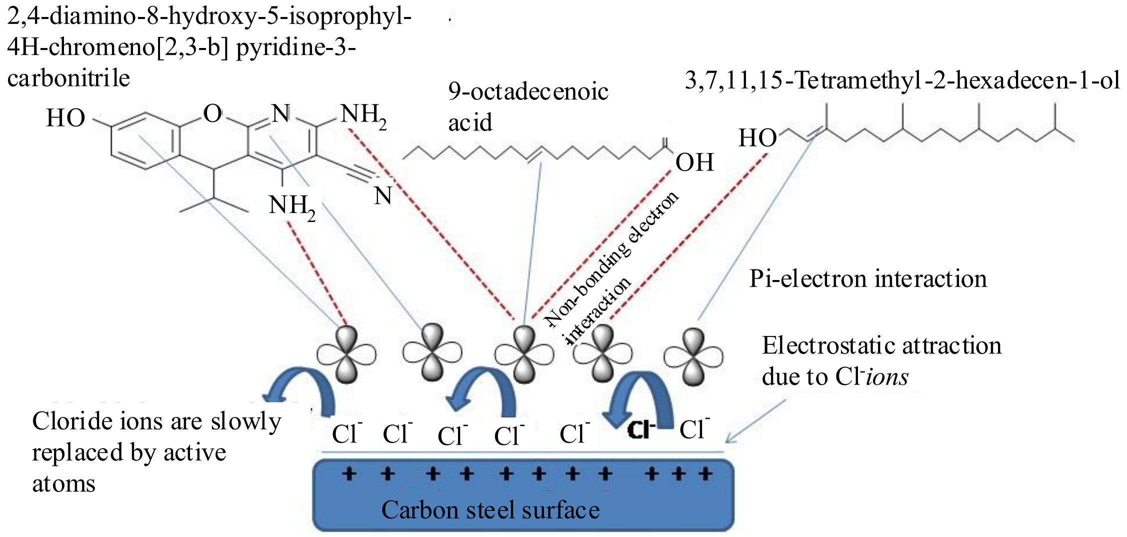

Figure 9. Mechanism proposed for system 1 and 2.

as the primary active component in systems 1 and 2. It is characterized by the presence of hydroxyl groups and olefinic double bonds that facilitate inhibition. 2,4-Diamino-8-hydroxy-5-isopropyl-5H-chromeno [2, 3-b] pyridine-3-carbonitrile is another important chemical in system 2 that is involved in corrosion inhibition. By making it easier for heteroatoms like nitrogen to interact with the carbon steel surface's vacant d-orbital, corrosion is successfully prevented [3, 7, 10]. As a consequence, system 1's maximum IE of 82% is surpassed. The possible process via which systems 1 and 2 impair performance on the steel surface is exposed in Figure 9.

CONCLUSIONS

This comparative study evaluated the inhibitive effects of System 1 and System 2 on carbon steel in acidic medium, yielding the following conclusions: System 2 (AIRE+ST) exhibited higher maximum inhibition efficiency (IE) of 93% compared to System 1's 82% at a concentration of 50+20 ppm. Increasing the concentration of both systems enhanced IE% while reducing corrosion rate (CR). The analysis of PDP curves revealed that both systems functioned as mixed-type inhibitors. GC-MS and FTIR analysis identified the active components responsible for protecting the carbon steel surface. SEM and AFM images revealed that the presence of System 1 and System 2 minimized surface morphological changes during corrosion. The superior performance of System 2 was attributed to the presence of heteroatoms like oxygen and nitrogen, making it a more effective mixed inhibitor for preventing carbon steel corrosion. Using systems 1 and 2, the thermodynamic and kinetic studies of corrosion will be carried out in the future.

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