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Electrochemical Investigation of the Adsorptive and Inhibitive Effects of Halide Ions and *Anacardium occidentale* Extracts on Mild Steel Corrosion in 1 M Sulphuric Acid

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Abstract

By utilizing electrochemical technique, the corrosion inhibition efficiency of Anacardium occidentale leave extracts on Grade 304 austenitic mild steel in 1 M sulphuric acid solution was examined. The crude extract of Anacardium occidentale leaves was used in this study to screened the phytochemicals and examine its inhibitory qualities as a corrosion inhibitor for mild steel in 1M sulphuric acid media. The phytochemicals showed the existence of many phytochemicals were flavonoids and alkaloid provide the majority.The efficacy of Anacardium occidentale leave extracts inhibition increased with concentration while decreasing with temperature. At 0.7 g/L extract concentration for 3 hours of immersion, the inhibitor's greatest efficiency was 94%. Mild steel corrosion was successfully inhibited by Anacardium occidentale leave extracts, according to the results obtained, and it was discovered that the presence of halide ions enhanced the efficacy of the inhibition. The synergistic effect of halide ions was found to follow the order: KI >KBr>KCl. According to the electron impedence spectroscopy (EIS), Anacardium occidentale functions as a mixed-type inhibitor. Due to the extract components' adsorption on the mild steel surface, leave extracts exhibit an inhibitory effect. The activation energy of the corrosion reaction increases by the presence of both extract and halide ion. The results of a study using scanning electron microscopy to examine the surface morphology of mild steel in inhibited and uninhibited acid solutions revealed that the presence of extract and halide inhibitors remarkably lowers the corrosion rate

Keywords: Anacardium occidentale leaves, Corrosion, Phytochemical, Inhibitor.

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INTRODUCTION

To meet the challenging requirements, mild steels are a class of adaptable materials that can be designed to display a wide range of engineering features through alloy design and regulated medical treatment [1]. This adaptability has resulted in increased demand for mild steel in a wide range of applications, including building construction, pipeline construction, automobile and machinery etc. because of its advantages of low cost, lightness and good corrosion resistance at moderate temperatures [2]. Dilute acid solutions are widely used in several industrial processes such as pickling, cleaning and descaling to remove the undesirable scales and rusts on the steel surface [3].

The active phytochemicals in plants that are effective for corrosion inhibition can be regarded as those with heteroatom in their aromatic or long chain, possession of π-electrons or suitable groups may also facilitate the transfer of charge from the inhibitor molecules to the charge metal surface (physical adsorption) or the transfer of electron from the inhibitor molecule to vacant d-orbital of the metal (chemical adsorption) [4]. Therefore, to identify the active constituents responsible for corrosion inhibition, a deeper understanding of the chemical structures and phytochemical composition of the plant extract is essential [5]. Plant extracts are rich sources of natural chemical compounds that can be isolated through simple, cost-effective methods and are inherently biodegradable [6]. As an environmentally friendly alternative to toxic and hazardous synthetic inhibitors, plant extracts contain a diverse range of organic compounds, including amino acids, alkaloids, steroids, flavonoids, proteins, and tannins, many of which contribute to their corrosion inhibitive properties [7].The molecular structure of corrosion inhibitors determines their effectiveness. So many authours like [8-10] have stated that organic corrosion inhibitors have heterogeneous atoms such as O, N, S, and P, which have high basicity and electron density, assisting in the corrosion inhibition of metals and alloys [11]. Since many corrosion inhibitors threaten the environment with their toxicity even though they possess high corrosion inhibition efficiency [12], this sparked interest among corrosion engineers and scientists also chemists, and polymer chemists and engineers to develop a new class of inhibitors that does not or pose minimal threat to the environment and the inhibitors should have high corrosion efficiency [13].

Anacardium occidentale is a plant with therapeutic qualities in all of its parts. *Anacardium occidentale leaves* which are frequently discarded, are an excellent source of vital nutrients, including dietary fibre and improves diet-related illnesses such as diabetes [14-15], antibacterial, antioxidant, and anti-inflammatory properties. The phytochemical components of the plant that have been examined, including flavonoids, alkaloids, phenols, steroids, saponins, tannins, carbohydrates, anthraquinolones, and glycoside. *Anacardium occidentale* leave extracts can be employed as a green corrosion inhibitor [16].

Anacardium occidentale leave extracts have been utilized as a corrosion inhibitor for different metals [7]. It has not, however, been studied as a potential mild steel inhibitor in an extremely corrosive environment such as 1 M H_2SO_4. Consequently, this study investigated the corrosion inhibition efficiency of

Anacardiumoccidentale leaf extract on mild steel in 1 M H_2SO_4 solution.

MATERIALS AND METHOD

Preparation of the leave Extracts

The extract was made by soaking 150 g of *Anacardium occidentale* leaves in 500 ml of 95% v/v ethanol for 48 hours. The mixture was first filtered through a muslin towel. Whatman No. 1 filter paper was then used to filter the resultant liquid, and the filtrate was concentrated using a rotary evaporator until a semi-solid extract was left [17]. The semi-solid extract obtained was oven-dried to a solid residue at 45 ^{0}C for 15 mins, weighed, and stored in a Bama bottle for use [18].

Phytochemical Analysis of *Anacardium occidentale* Leaves Extract

Phytochemical examinations were carried out for *Anacardium occidentale* leaves extracts as per the standard methods described by many authors.

Detection of Steroid: One gram (1g) of each extract was diluted in one centilitre of ethanol. The solution was then supplemented with 1 cm^3 of concentrated H_2SO_4. The development of the red colouring suggested the presence of steroids [19].

Detection* of *Alkaloid: The presence of alkaloids was determined by dissolving each extract separately in diluted HCl, followed by filtration of the resulting solution.The presence of alkaloids was determined by dissolving each extract separately in diluted HCl, followed by filtration of the resulting solution.

Mayer's Test: Mayer's reagent (potassium mercuric iodide) was applied to the filtrates. Formation of a yellow-colored precipitate indicates the presence of alkaloids [20].

Wagner's Test: Iodine in Potassium Iodide, Wagner's reagent, was applied to the filtrates. Formation of brown/reddish precipitate indicates the presence of alkaloids [21].

Detection of Anthraquinolones*:* 10cm^3 of benzene was introduced to 5g of the plant extract, shaken and filtered. 5cm^3 of 10% NH_3 solution was then be added to the filtrate. The mixture was shaken and the formation of a pink or violet colour indicates the presence of anthraquinolones [22].

Detection of Glycoside*:* After extracts were hydrolysed with dil. HCl, they were tested for glycosides.

Modified Borntrager's Test: the extract was treated with a solution of ferric chloride and submerging them in boiling water for roughly five minutes. After cooling, the mixture was extracted using the same amount of benzene. After being separated, the benzene layer was treated with an ammonia solution. Formation of rose-pink color in the ammonical layer indicates the presence of anthranol glycosides [16].

Detection of Saponins*:* The extracts were shaken in a graduated cylinder for 15 minutes after being diluted with 20 millilitres of distilled water. There are saponins present when a 1 cm layer of forms [23].

Foam Test: 0.5gm of extract was diluted with 2ml of water. A foam produced persists for ten minutes, indicating the presence of saponins [24].

Detection of Phenols*:* Ferric Chloride Test: the Extracts were treated with 3-4 drops of Ferric Chloride solution. Formation of a bluish black color indicates the presence of phenols [25].

***Detection of Tannins**:* Gelatin Test: To the extract, 1% gelatin solution containing Sodium Chloride was added. Formation of a white precipitate indicates the presence of tannins. [24].

Detection of Flavonoids

Alkaline Reagent Test: A few drops of sodium hydroxide solution were added to the extracts. Flavonoids are indicated by the formation of a bright yellow colour that turns colourless when diluted acid is added.

Lead Acetate Test: A few drops of lead acetate solution were added to the extracts. When a yellow-colored precipitate forms, flavonoids are present [25].

Detection of proteins and amino acids

Xanthoproteic Test: A few drops of concentrated HNO_3 acid were added to the extracts. The development of a yellow hue signifies the existence of proteins.

Ninhydrin Test: The extract was heated for a few minutes after 0.25% w/v ninhydrin reagent was added. The presence of amino acids is shown by the formation of a blue colour [26].

Detection of Carbohydrate: Each extract was separately diluted in 5ml of distilled water and then filtered. The presence of carbohydrates was tested using the filterate.

Molisch's Test: In a test tube, filtrates were exposed to two drops of an alcoholic α-naphthol solution. Carbohydrates are present when the violet ring forms at the intersection [27].

Benedict's test: Filtrates were diluted with Benedict's reagents and gently heated. Orange red precipitate, indicates the presence of reducing sugars [6].

Coupon Preparation

Commercially sourced mild steel was cut into coupons measuring 9 × 3 cm. The coupons were mechanically polished using 400-grade emery paper, then cleaned with acetone, degreased in ethanol, rinsed with distilled water, and air-dried. They were They were subsequently stored in a desiccator for 15 minutes [5]. The chemical composition of the mild steel was determined using an Optical Emission Spectrophotometer (OES) , yielding the following (wt. %): P 0.09, Si 0.38, Al 0.01, Mn 0.05, C 0.21, S 0.05, and Fe (balance) 99.42% [28].

Electrochemical studies

Electrochemical experiments were conducted using a computer-controlled Parstat 2273 potentiostat. Data acquisition was performed with PowerSuite software, and analysis was carried out using ZsimpWin software (version 3.21). A three-electrode setup was employed, consisting of a platinum foil as the auxiliary electrode, a saturated calomel electrode (SCE) as the reference electrode, and a mild steel coupon prepared as described in the weight loss method as the working electrode. Measurements were taken at open circuit potential (OCP) after 30 minutes of immersion in the corrosive medium, by superimposing a sinusoidal AC signal of 10 mV amplitude over a frequency range of 10^6 to 10^{-2} Hz. The double-layer capacitance (Cdl) and charge transfer resistance (Rct) were obtained from the Nyquist plots. The inhibition efficiency (IE) was calculated using the corresponding relationship [29].

$$IE = \frac{R'ct - Rc}{R'ct} \text{ x } 100 \quad (1)$$

Where R_{ct} and R'_{ct} are the charge transfer resistance values in the absence and presence of inhibitors, respectively [4].

The Cdl value was calculated from the following equation.

$$\text{Cdl} = \frac{1}{(w+Rct)} \text{ x } 100 \quad (2)$$

SEM analysis

Scanning Electron Microscope (SEM) analysis was used to study the metal surfaces after 3 hours' immersion time to understand the changes that occur before and after corrosion in the presence and absence of the extracts and halide ions using the Supra 40VP model [18]

RESULTS AND DISCUSSION

Table 1: Phytochemicals of Anacardium occidentale Leave Extracts In The Presence Of Ethan

S/N	Chemical Category	Name Of Test	Ethanol Extract
1	Alkaloids	Wagner's test	+
2	Glycosides	Borntrager's test	+
3	Flavonoids	Alkaline reagent test	+
4	Carbohydrates	Molisch's test	+
5	Proteins andAmino Acid	Xanthoprotein test	_
6	Tannins	Potassium	+
7	Saponins	dichromate	+
8	Phenols	Froth test	+
9	Anthraquinolones	Ferric chloride test	+
10	Steroid	Benzene Conc. H_2SO_4 test	+

Key: + = Present; - = Absent

The qualitative phytochemical results showed the presence and absence of certain phytochemicals in the extracts in (Tables 1). The test was performed using ethanol to extract the leaves of the plants.

The preliminary phytochemical screening of *Anacardium occidentale* revealed the presence of carbohydrates, glycoside, tannins, anthraquinolones, phenols, steroids, alkaloids, flavonoids and saponins while protein and amino acid are absent. The presence of phytochemicals makes plants effective as corrosion inhibitors. The leaves of the plant help reduce the corrosion rate through several mechanisms: (i) adsorption of ions or molecules onto the metal surface, (ii) modulation of anodic and/or cathodic reactions, (iii) reduction in the diffusion rate of reactants to the metal surface, and (iv) alteration of the electrical resistance at the metal interface. These green inhibitors are non-toxic to humans and environmentally friendly [16].

Table 2. Electrochemical Impedance Spectroscopy (E.I.S) of *Anacardium occidentale* leave extracts withiout halide ions for different concentrations and temperatures

Temperature (^{0}C)	Concentration (g/L)	Charge transfer resistace (R'ct&Rct) (µcm^2)	Double layer capacitance (Cdl)	Inhibition efficiency (%IE)	Surface Coverage (θ)
30 (^{0}C)	BLANK	62.00	2.20×10^{-3}	-	-
	0.3g	152.4	9.80×10^{-4}	59	0.590
	0.5g	163	9.20×10^{-4}	61	0.610
	0.7g	192	7.80×10^{-4}	67	0.670
60 (^{0}C)	0.3g	106.5	1.36×10^{-3}	41	0.410
	0.5g	103	1.40×10^{-3}	39	0.390
	0.7g	109	1.33×10^{-3}	43	0.430
90 (^{0}C)					
	0.3g	81.60	1.90×10^{-4}	24	0.240
	0.5g	92.21	1.50×10^{-3}	32	0.320
	0.7g	97.7	1.47×10^{-3}	36	0.360

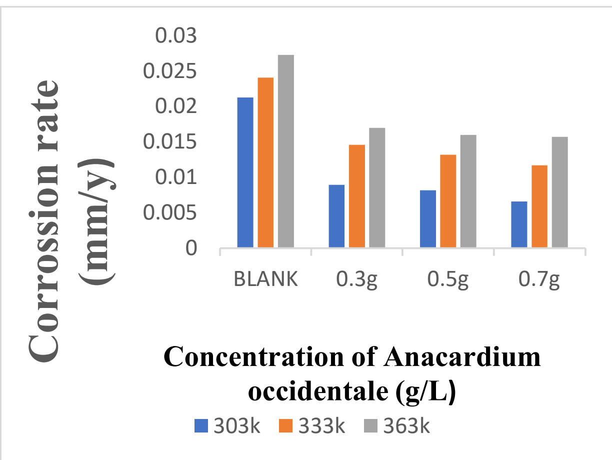


Figure 1: Corrosion Rate of Mild Steel using AO at different temperature

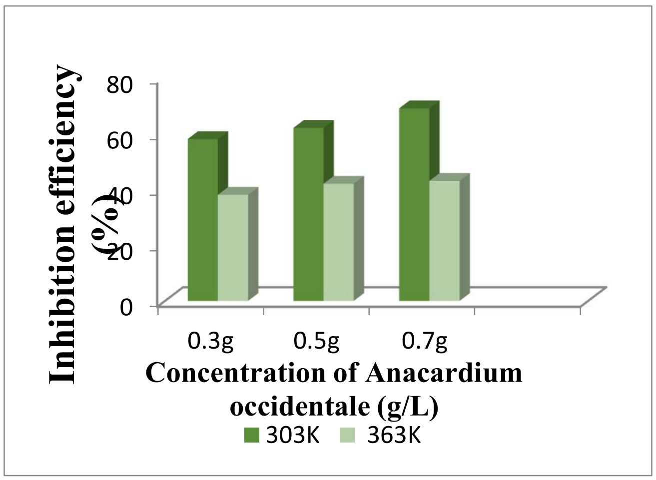


Figure 2: Inhibition efficiency of AO At different temperature

Table 3. Electrochemical Impedance Spectroscopy (E.I.S) of *Anacardium occidentale* leave extracts with halide ions for different concentrations and temperatures

Temperature (^0C)	Concentration (g/L)	Charge transfer resistace (R'ct&Rct) (μcm^2)	Double layer capacitance (Cdl)	Inhibition efficiency (%IE)	Surface Coverage (θ)
30 (^{0}C)	BLANK	53.56	2.5×10^{-3}	-	-
	0.3 in KCl	161.7	9.3×10^{-4}	66.0	0.660
	0.3 in KBr	182.4	8.33×10^{-4}	70.0	0.700
	0.3 in KI	272.6	5.66×10^{-4}	80.0	0.800
	0.5 in KCl	168.2	9.00×10^{-4}	68.0	0.680
	0.5 in KBr	206.0	7.41×10^{-4}	74.0	0.740
	0.5 in KI	307.8	5.03×10^{-4}	82.0	0.820
	0.7 in KCl	198.9	7.67×10^{-4}	73.0	0.730
	0.7 in KBr	365.2	4.25×10^{-4}	85.0	0.850
	0.7 in KI	902.0	1.74×10^{-4}	94.0	0.940
60 (^{0}C)	0.3 in KCl	101.6	1.44×10^{-3}	47.0	0.470
	0.3 in KBr	113.8	1.30×10^{-3}	52.0	0.520
	0.3 in KI	138.7	1.08×10^{-3}	61.0	0.610
	0.5 in KCl	108.7	1.35×10^{-3}	50.0	0.500
	0.5 in KBr	127.4	1.17×10^{-3}	57.0	0.570
	0.5 in KI	184.6	8.24×10^{-4}	82.0	0.820
	0.7 in KCl	133.9	1.11×10^{-3}	60.0	0.600
	0.7 in KBr	137.8	1.08×10^{-3}	61.0	0.610
	0.7 in KI	204.5	7.47×10^{-4}	73.0	0.730
90 (^{0}C)	0.3 in KCl	088.8	1.63×10^{-3}	39.0	0.390
	0.3 in KBr	103.4	1.42×10^{-3}	48.0	0.480
	0.3 in KI	120.2	1.23×10^{-3}	55.0	0.550
	0.5 in KCl	107.6	1.37×10^{-3}	50.0	0.500
	0.5 in KBr	115.3	1.28×10^{-3}	53.0	0.530
	0.5 in KI	128.1	1.16×10^{-3}	58.0	0.580
	0.7 in KCl	095.5	1.52×10^{-3}	43.0	0.430
	0.7 in KBr	121.9	1.22×10^{-3}	56.0	0.560
	0.7 in KI	172.5	8.79×10^{-4}	68.0	0.680

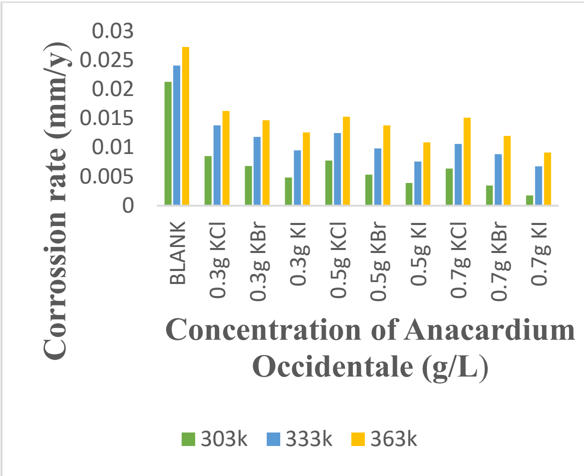


Figure 1: Corrosion Rate of Mild Steel using AO and halide at different temperature

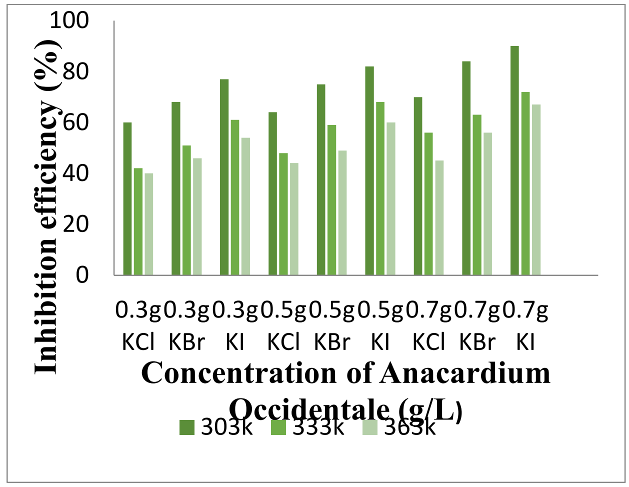
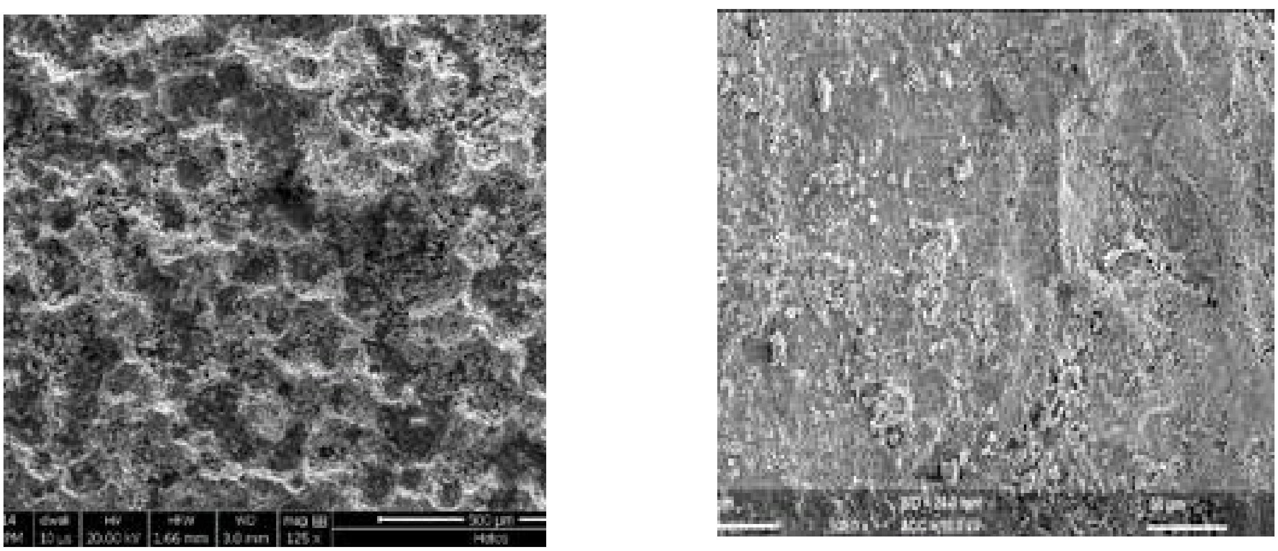


Figure 2: Inhibition efficiency of AO and halide at different temperature

The measurements were undertaken to assess the interactions of the mild steel/electrolyte interface in the presence and absence of *AnacardiumOccidentale* with different concentrations of halide ions. The impedance data for mild steel in 0.1 M H_2SO_4 with and without *AnacardiumOccidentale* and in combination with KCl, KBr and KI were recorded and listed in (Tables 2 and 3).

The electron impedence spectroscopy findings indicated that the inhibitory efficiency rose with concentration. The greatest inhibitory efficacy of KI in *Anacardium occidentalis* was 94% at 0.7 g/L. This implies that extracts from *Anacardium occidentalis* are quite effective at preventing corrosion [30]. This is accredited to the absorption of nutrients of the extracts on the surface of the mild steel which makes a barrier for mass and charge transfer and prevents further corrosion [23]. According to the results presented in Tables 2 and 3, both the plant extracts and halide ions are effective inhibitors of mild steel corrosion in 1 M H_2SO_4 at 30 °C, with inhibition efficiency declining at higher or lower temperatures. The highest inhibition efficiency of 67% was observed at 30 °C with 0.7 g/L of *Anacardium occidentale* extract. This finding is consistent with previous studies [3]. The study also demonstrated that the addition of halide ions—specifically KI, KBr, and KCl—to the acidic solution containing the extract significantly enhanced the inhibition performance compared to the extract alone. When 0.3 g/L of KI, KBr, or KCl was combined with 0.7 g/L of extract in 1 M H_2SO_4, the inhibition efficiency increased to 94%.The type of halide ion used significantly influenced the inhibition efficiency, with the greatest synergistic effect observed at the highest concentration of each halide. Among the halides, iodide exhibited the strongest synergistic effect, which aligns with findings reported in previous studies [12]. Analysis of the electrochemical parameters revealed that the introduction of *Anacardium occidentale* extract into the acidic medium led to an increase in charge transfer resistance (Rct) and a corresponding decrease in double-layer capacitance (Cdl). These effects became more pronounced when the extract was used in combination with halide ions [31]. The observed decrease in Cdl is typically attributed to a reduction in the local dielectric constant and/or an increase in the thickness of the electrical double layer both of which are indicative of enhanced surface coverage by the inhibitor. This behaviour confirms the adsorption of the extract and halide ions onto the metal/electrolyte interface. The increase in Rct and decrease in Cdl values, as shown in Tables 2 and 3, can be ascribed to the displacement of water molecules by *Anacardium occidentale* components and halide ions on the electrode surface, leading to the formation of a protective barrier and a thicker double layer. As a result, the inhibition efficiency of *Anacardium occidentale* increased from 67% to 73% with the addition of KCl, 85% with KBr, and reached a maximum of 94% with KI. These findings are consistent with those reported in previous studies [30]. The highest inhibition efficiency was achieved with the combination of *Anacardium occidentale* leaf extract and potassium iodide, demonstrating its strong synergistic effect and high effectiveness as a corrosion inhibitor for mild steel in acidic media. Additionally, this green inhibitor system offers an environmentally friendly alternative to conventional corrosion inhibitors.

Morphological Properties of Corrosion Study using SEM analysis



(a) (b)

Plate1. SEM micrographs of Mild steels after corrosion in 1 M H_2SO_4 containing both extracts at 30^0C (a) Blank (b) 0.7 g/L *Anacardium occidentale.*

SEM was used to examine the surface morphology of a few chosen samples. The blank sample plates surface is severely corroded, as seen in the micrographs plate 1a. As the temperature rises, the corrosion agent harshness on the metal surface in blank solution that is a solution free of inhibitors increases. After three hours of immersion in a 1M concentrated H_2SO_4 solution at 30°C, these pictures verified the rates at which the metal corroded. The micrographs in plate 1.b showed that mild steel surfaces were protected from corrosion and that smoother surfaces arise as the inhibitor concentration increased. This is in support of findings from [9].

CONCLUSION

The extract of *Anacardium occidentale* leaves contains a variety of phytochemicals, including carbohydrates, steroids, tannins, alkaloids, glycosides, phenols, flavonoids, and saponins. Among these, flavonoids were the most abundant, followed by alkaloids.

Anacardium occidentale leaf extract acts as an effective green corrosion inhibitor for mild steel in 1 M H_2SO_4 solution. The inhibition efficiency increases with increasing extract concentration.

The extract functions as a mixed-type inhibitor, influencing both anodic and cathodic reactions.

The adsorption of *Anacardium occidentale* on the mild steel surface follows a physical adsorption mechanism.

Electrochemical Impedance Spectroscopy (EIS) demonstrated a significant inhibition efficiency of 94% when combined with potassium iodide (KI), and 67% without halide addition.

Surface analysis via Scanning Electron Microscopy (SEM) confirmed the protective film formation by the extract on the steel surface, supporting its inhibitory action.

Therefore, the phytochemicals present in *Anacardium occidentale* leaf extract can effectively inhibit mild steel corrosion in 1 M H_2SO_4.

The inhibition performance is influenced by factors such as extract concentration, temperature, and exposure time and these dependencies were accurately described by a quadratic model.

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CONFLICT OF INTEREST

Authours declared no conflict of interest.

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ETHICAL STATEMENT

This work required no ethical statement.

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