



# 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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Received: 10<sup>th</sup> May, 2025

Revised: 21<sup>st</sup> June, 2025

Accepted: 7<sup>th</sup> August, 2025

## 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 impedance 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.

## 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  $\pi$ -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 authors 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

*Anacardium occidentale* leaf extract on mild steel in 1 M H<sub>2</sub>SO<sub>4</sub> 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 °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<sup>3</sup> of concentrated H<sub>2</sub>SO<sub>4</sub>. 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<sup>3</sup> of benzene was introduced to 5g of the plant extract, shaken and filtered. 5cm<sup>3</sup> of 10% NH<sub>3</sub> 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<sub>3</sub> 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 filtrate.

**Molisch's Test:** In a test tube, filtrates were exposed to two drops of an alcoholic  $\alpha$ -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 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<sup>6</sup> to 10<sup>-2</sup> Hz. The double-layer capacitance (Cdl) and charge transfer resistance (R<sub>ct</sub>) were obtained from the Nyquist plots. The inhibition efficiency (IE) was calculated using the corresponding relationship [29].

$$IE = \frac{R'_{ct} - R_{ct}}{R'_{ct}} \times 100 \quad (1)$$

Where R<sub>ct</sub> and R'<sub>ct</sub> are the charge transfer resistance values in the absence and presence of inhibitors, respectively [4].

The Cdl value was calculated from the following equation.

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

### 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 and Amino Acid	Xanthoprotein test	—
6	Tannins	Potassium	+
7	Saponins	dichromate	+
8	Phenols	Froth test	+
9	Anthraquinolones	Ferric chloride test	+
10	Steroid	Benzene	+
		Conc. H <sub>2</sub> SO <sub>4</sub> test	

**Key:** + = Present; - = Absent

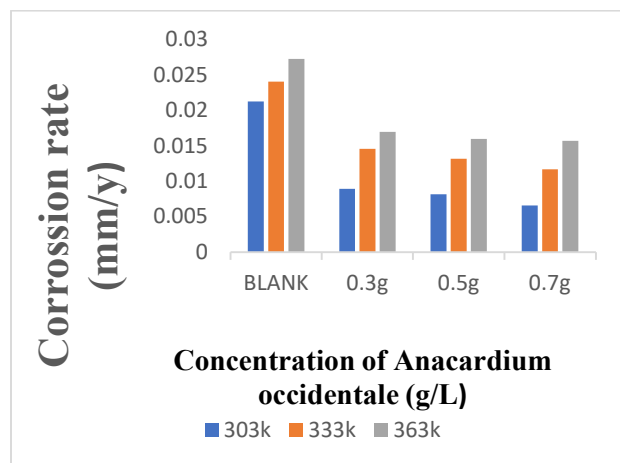
The qualitative phytochemical results showed the presence and absence of certain phytochemicals in the extracts in (Tables 1). The test was performed using ethanol to extract the leaves of the plants.

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

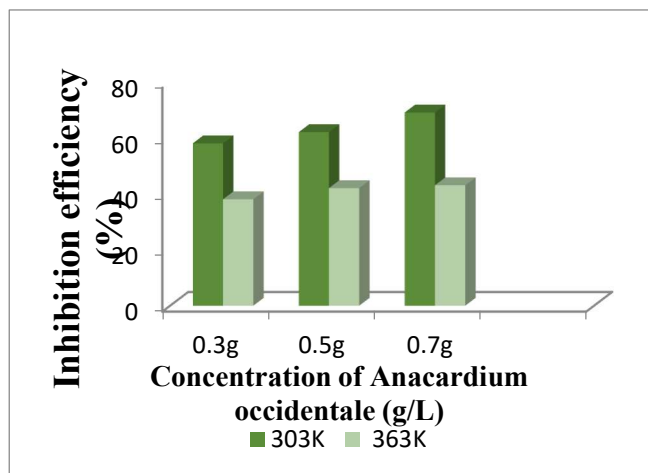
of ions or molecules onto the metal surface, (ii) modulation of anodic and/or cathodic reactions, (iii) reduction in the diffusion rate of reactants to the metal surface, and (iv) alteration of the electrical resistance at the metal interface. These green inhibitors are non-toxic to humans and environmentally friendly [16].

**Table 2.** Electrochemical Impedance Spectroscopy (E.I.S) of *Anacardium occidentale* leave extracts without halide ions for different concentrations and temperatures

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



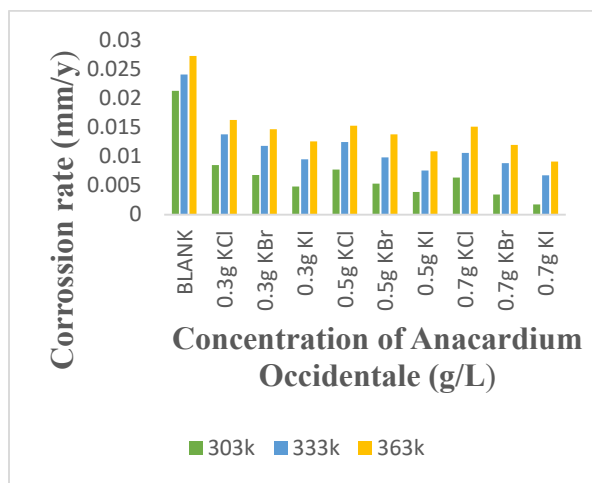
**Figure 1:** Corrosion Rate of Mild Steel using AO at different temperature



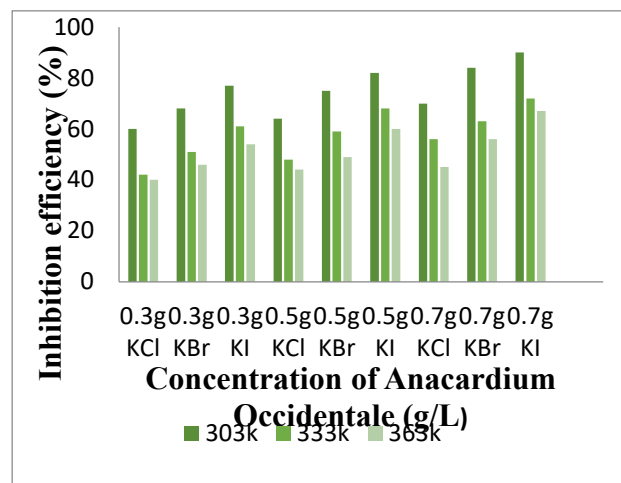
**Figure 2:** Inhibition efficiency of AO At different temperature

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

Temperature ( $^{\circ}\text{C}$ )	Concentration (g/L)	Charge transfer resistance ( $R'_{ct} \& R_{ct}$ ) ( $\mu\text{cm}^2$ )	Double layer capacitance (Cdl)	Inhibition efficiency (%IE)	Surface Coverage ( $\theta$ )
30 ( $^{\circ}\text{C}$ )	BLANK	53.56	$2.5 \times 10^{-3}$	-	-
	0.3 in KCl	161.7	$9.3 \times 10^{-4}$	66.0	0.660
	0.3 in KBr	182.4	$8.33 \times 10^{-4}$	70.0	0.700
	0.3 in KI	272.6	$5.66 \times 10^{-4}$	80.0	0.800
	0.5 in KCl	168.2	$9.00 \times 10^{-4}$	68.0	0.680
	0.5 in KBr	206.0	$7.41 \times 10^{-4}$	74.0	0.740
	0.5 in KI	307.8	$5.03 \times 10^{-4}$	82.0	0.820
	0.7 in KCl	198.9	$7.67 \times 10^{-4}$	73.0	0.730
	0.7 in KBr	365.2	$4.25 \times 10^{-4}$	85.0	0.850
	0.7 in KI	902.0	$1.74 \times 10^{-4}$	94.0	0.940
60 ( $^{\circ}\text{C}$ )	0.3 in KCl	101.6	$1.44 \times 10^{-3}$	47.0	0.470
	0.3 in KBr	113.8	$1.30 \times 10^{-3}$	52.0	0.520
	0.3 in KI	138.7	$1.08 \times 10^{-3}$	61.0	0.610
	0.5 in KCl	108.7	$1.35 \times 10^{-3}$	50.0	0.500
	0.5 in KBr	127.4	$1.17 \times 10^{-3}$	57.0	0.570
	0.5 in KI	184.6	$8.24 \times 10^{-4}$	82.0	0.820
	0.7 in KCl	133.9	$1.11 \times 10^{-3}$	60.0	0.600
	0.7 in KBr	137.8	$1.08 \times 10^{-3}$	61.0	0.610
	0.7 in KI	204.5	$7.47 \times 10^{-4}$	73.0	0.730
90 ( $^{\circ}\text{C}$ )	0.3 in KCl	088.8	$1.63 \times 10^{-3}$	39.0	0.390
	0.3 in KBr	103.4	$1.42 \times 10^{-3}$	48.0	0.480
	0.3 in KI	120.2	$1.23 \times 10^{-3}$	55.0	0.550
	0.5 in KCl	107.6	$1.37 \times 10^{-3}$	50.0	0.500
	0.5 in KBr	115.3	$1.28 \times 10^{-3}$	53.0	0.530
	0.5 in KI	128.1	$1.16 \times 10^{-3}$	58.0	0.580
	0.7 in KCl	095.5	$1.52 \times 10^{-3}$	43.0	0.430
	0.7 in KBr	121.9	$1.22 \times 10^{-3}$	56.0	0.560
	0.7 in KI	172.5	$8.79 \times 10^{-4}$	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 *Anacardium Occidentale* with different concentrations of halide ions. The impedance data for mild steel in 0.1 M  $H_2SO_4$  with and without *Anacardium Occidentale* and in combination with KCl, KBr and KI were recorded and listed in (Tables 2 and 3).

The electron impedance 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

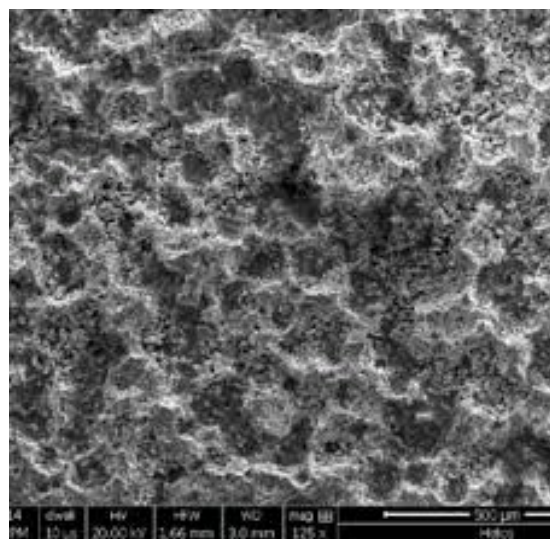
corrosion in 1 M  $H_2SO_4$  at 30 °C, with inhibition efficiency declining at higher or lower temperatures. The highest inhibition efficiency of 67% was observed at 30 °C with 0.7 g/L of *Anacardium occidentale* extract. This finding is consistent with previous studies [3]. The study also demonstrated 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*



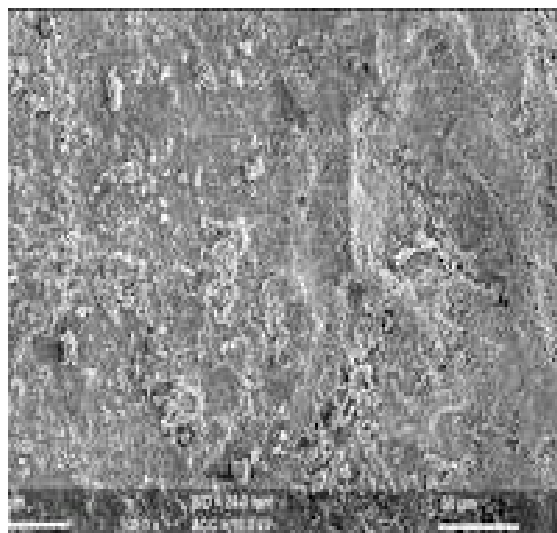
extract into the acidic medium led to an increase in charge transfer resistance ( $R_{ct}$ ) and a corresponding decrease in double-layer capacitance ( $C_{dl}$ ). These effects became more pronounced when the extract was used in combination with halide ions [31]. The observed decrease in  $C_{dl}$  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  $R_{ct}$  and decrease in  $C_{dl}$  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°C (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.

## ACKNOWLEDGEMENTS

The supports and assistance of the entire laboratory and the botanist staff are highly commendable.

## CONFLICT OF INTEREST

Authors declared no conflict of interest.

## FUNDING

There was no funding received for this work.

## ETHICAL STATEMENT

This work required no ethical statement.

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# Determination of Heavy Metals Content and Physico-Chemical Parameters in Soil from Tsamawa, Kaita and Kofar Sauri Irrigation Sites

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Received: 4<sup>th</sup> May, 2025

Revised: 2<sup>nd</sup> July, 2025

Accepted: 7<sup>th</sup> August, 2025

## Abstract

This research determined heavy metals (Cu, Fe, Cd, Cr, Pb, Ni, Mn, Zn) and physicochemical parameters in soil samples from irrigation sites in Tsamawa (Kano), Kaita, and Kofar Sauri in Katsina. Soil pH ranged from 6.05 in Tsamawa (KISS) to 6.30 in Kofar Sauri (KUSS). Water holding capacity varied between 40.65% in KUSS to 75.53% in Kaita (KSS), while cation exchange capacity ranged from 4.33 Cmol/kg (KUSS) to 6.43 Cmol/kg (KSS). Organic carbon was highest in KSS (2.43), followed by KISS (1.62), and lowest in KUSS (1.39). Organic matter values were 4.20 (KSS), 2.80 (KISS), and 2.40 (KUSS). Soil textures were clay-loam (KSS: 52.2% clay, 32.4% sand, 15.4% silt), sandy-clay-loam (KISS: 62.0% sand, 22.3% clay, 15.7% silt), and sandy-loam (KUSS: 70.3% sand, 14.4% clay, 13.8% silt). Heavy metal concentrations exceeded FAO/WHO limits for Cd, Cr, Mn, Ni, Pb, and Zn. KSS had the highest Cd (1.443 mg/kg), Cu (3.882 mg/kg), and Pb (1.749 mg/kg), while KUSS showed elevated Cr (2.948 mg/kg), Ni (1.193 mg/kg), and Zn (1.701 mg/kg). KISS had the lowest contamination. Fe and Cu remained within safe limits. The findings indicate potential health and environmental risks, requiring remediation and further investigation into pollution sources.

**Keywords:** Pollution, Heavy metals, Soil, Physico-chemical parameters.

## INTRODUCTION

Heavy metals are naturally occurring elements with atomic numbers greater than 20, known for their high density (at least 5 g/cm<sup>3</sup>) and toxicity even at low concentration [1, 2]. These elements are present in various amounts in the environment and are integral to many human activities. They are found in essential structures and a variety of artificial mixtures [3, 4]. Human activities have significantly altered the biochemical cycles and balance of many heavy metals, leading to their use in products like cars and batteries.

These metals come from both man-made and natural sources and eventually make their way into the environment [5]. Natural sources include volcanic eruptions and the weathering of metal-bearing rocks. Human activities such as the excessive use of chemical fertilizers, wood burning, coal combustion, vehicle emissions, mining, smelting, and incineration have disrupted natural metal cycles, resulting in significant accumulations of heavy metals, particularly in soil [6, 7]. The main heavy metals of concern include lead, cadmium, chromium, zinc, nickel, and manganese due to their toxic effects on human health. High environmental concentrations of these metals have been linked to various cancers and kidney problems [8, 9]. Moreover, heavy metals negatively affect soil microorganisms and plant growth and development [10, 11]. Their persistence and potential for harm have made them a significant environmental concern over recent decades. Besides being non-biodegradable, heavy metals can undergo microbial or chemical transformations [12, 13]. Recent studies by [14] and [15] indicate that environments polluted with heavy metals can activate processes that decrease microbial tolerance to antibiotics due to the

co-regulation of antibiotic resistance genes. Heavy metals can contaminate the environment through various pathways. Due to their stability, they can persist in environmental compartments long after initial deposition. Soil and water systems can also become polluted from the weathering of disposed products [16]. Accumulations of heavy metals in plants and soil from natural and artificial sources represent significant pollution problems. Food safety issues and potential health risks make this a serious environmental concern [17]. Some heavy metals, like copper, zinc, manganese, cobalt, and molybdenum, are micronutrients necessary for growth in trace amounts, while others, like cadmium, arsenic, and chromium, are carcinogenic [18]. Mercury and lead are linked to developmental abnormalities in children. Long-term cadmium exposure causes renal, prostate, and ovarian cancers [19]. Excessive levels of heavy metals can cause biochemical effects such as competition for binding sites with essential metabolites, replacement of essential ions, reactions with -SH groups, damage to cell membranes, and reactions with phosphate groups [20].

Irrigation system is known to contribute significantly to heavy metal contents of soils [21, 22]. Majority of crops and vegetables consumed in Kano and Katsina states were produced through irrigation, and mostly the water used by the farmers is waste water from urban or industrial discharges and other waste water channels (including contaminated wells and aquifers) [23, 24]. Therefore, there is need for periodic monitoring of these irrigation sites so as to take appropriate measures to minimize the accumulation of these metals in the soil. Physicochemical parameters analyses will

help in ascertaining the soil properties and features that might likely contribute to the accumulation of these metals, while the heavy metals analyses will give us the actual concentration of the metals in the soil whether low, high or within safe limits when compared with world health organization (FAO/WHO) safe limits and standards.

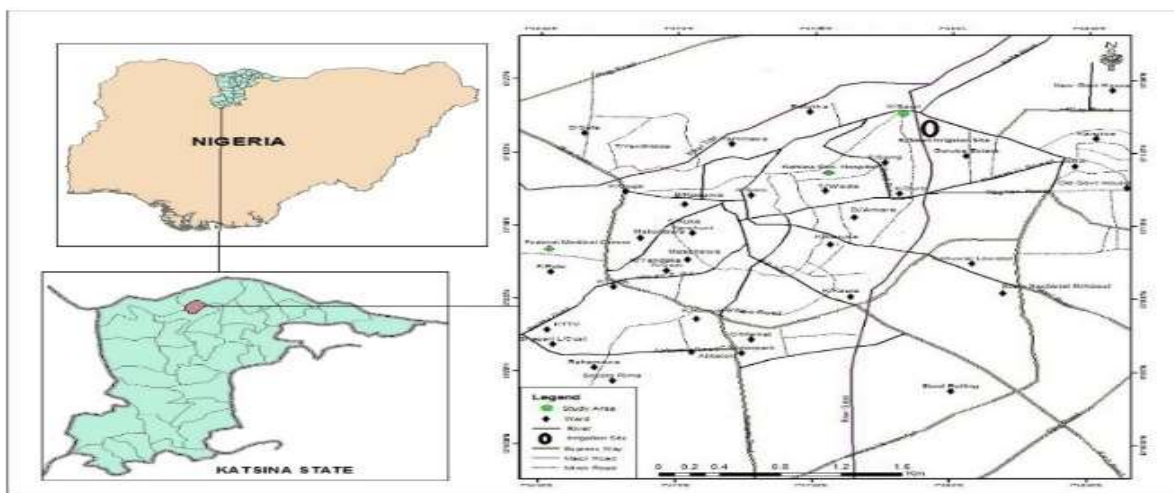
## MATERIALS AND METHODS

### Description of study areas

Kofar Sauri irrigation area is one of the many irrigation sites located in Katsina urban area, Katsina State. The irrigation area is located at the extreme northern margin of Nigeria, has a total land area of about 3,370 square kilometers and lies between latitudes  $11^{\circ}08'N$  and  $13^{\circ}22'N$  and longitude  $6^{\circ}52'E$  and  $9^{\circ}20'E$  [19] The Kofar Sauri sampling site has a predominantly ferruginous, tropical red and brown soil, underlain by basement complex rocks. Over large areas, the vegetation does not provide adequate cover for the soils especially at the beginning of the rains; hence the soils are generally susceptible to erosion. The climate is hot and dry for most of the year. Maximum day

temperature of about  $43^{\circ}C$  in the months of March, April and May are common and the minimum temperature is about  $22^{\circ}C$  in the month of December and January [20], while the mean annual rainfall is 780mm.

Kofar Sauri irrigation site is located along the major waste channel in a highly populated residential area of Katsina metropolis. The wastewater channel that provides water for irrigation to the farmers was previously a naturally flowing river, called River Ginzo, with a total drainage area of about  $6.4\text{ Km}^2$  and length of 48 Km [21]. The Channel was not contaminated before as it originates from higher areas around Dutsinma but gradually become contaminated due to human activities (domestic, industrial, mechanical, etc.). Substantial crop and vegetable production takes place on the right side of the road, and are being irrigated by the wastewater released from the residential areas [8], some small-scale tanneries, dye-pits, abattoir, car washing, welding, electroplating and painting spots are also located in certain parts of the city, where effluent are being discharged into the waste channel.



**Figure 1.** Map showing Kofar Sauri sample area in Katsina city, Nigeria

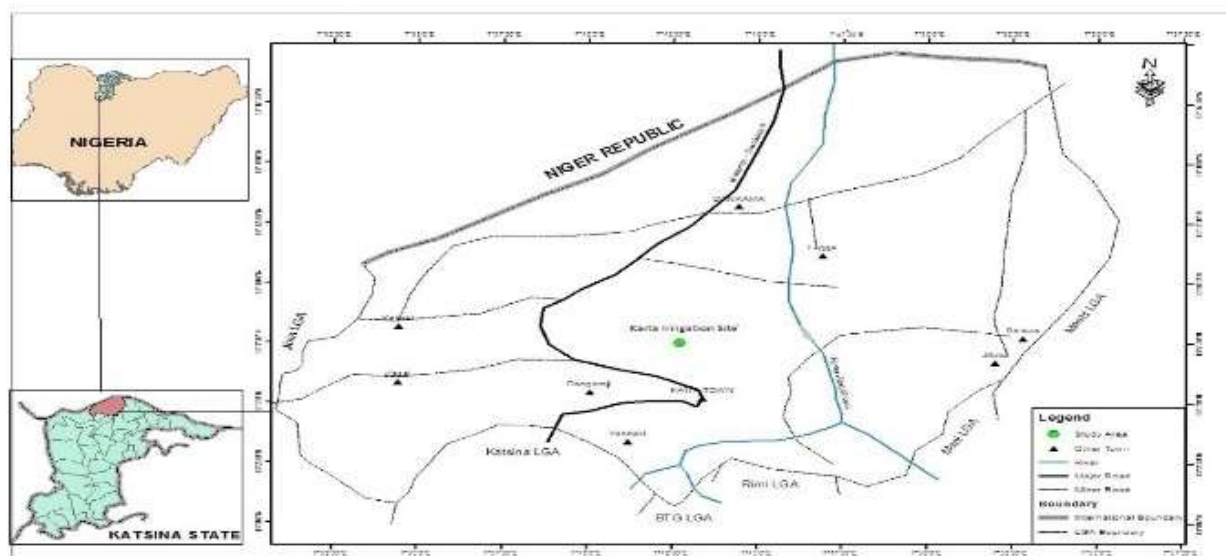


Kaita is a local government area in Katsina state, it is located in the northern part of the state near the border with Niger. The area is predominantly underlain by Precambrian basement complex rocks, including magnetite, granite gneiss and porphyritic granites. These crystalline formations are occasionally overlain by cretaceous sedimentary rocks of the Gundumi formations (sedimentary layer), which comprises coarse feldspathic grits interbedded with purplish clay and quartz pebbles [22].

The soils are primarily ferruginous tropical red and brown types derived from the weathering of basement rocks and sandy drifts; they are also susceptible to erosion. The basement complex rocks typically yield low ground water due to their crystalline nature, but areas underlain by Gundumi formations offer higher ground water potential due to porosity and permeability of the sedimentary rocks.

The basement complex rocks, the Gundumi formations containing feldspathic grits and quartz pebbles are often rich in naturally occurring heavy metals such as, Pb, Zn, Cu, Cd and As. When these rocks weather and breakdown, they release trace metals into the soil which can lead to elevated metal concentrations particularly in clay-rich soils. Ground water moving through the porous sedimentary layer of Gundumi formation can also mobilise and redistribute metals into the overlying soil [22,23]. Therefore naturally, metals are bound to be present in this irrigation area, and hence, might require remediation

Irrigation in Kaita is carried out using underground water (Tube wells) which the farmers dug in their respective farms. Rice is the major grain grown during rainy season, while wheat and many other vegetables are grown during dry season.



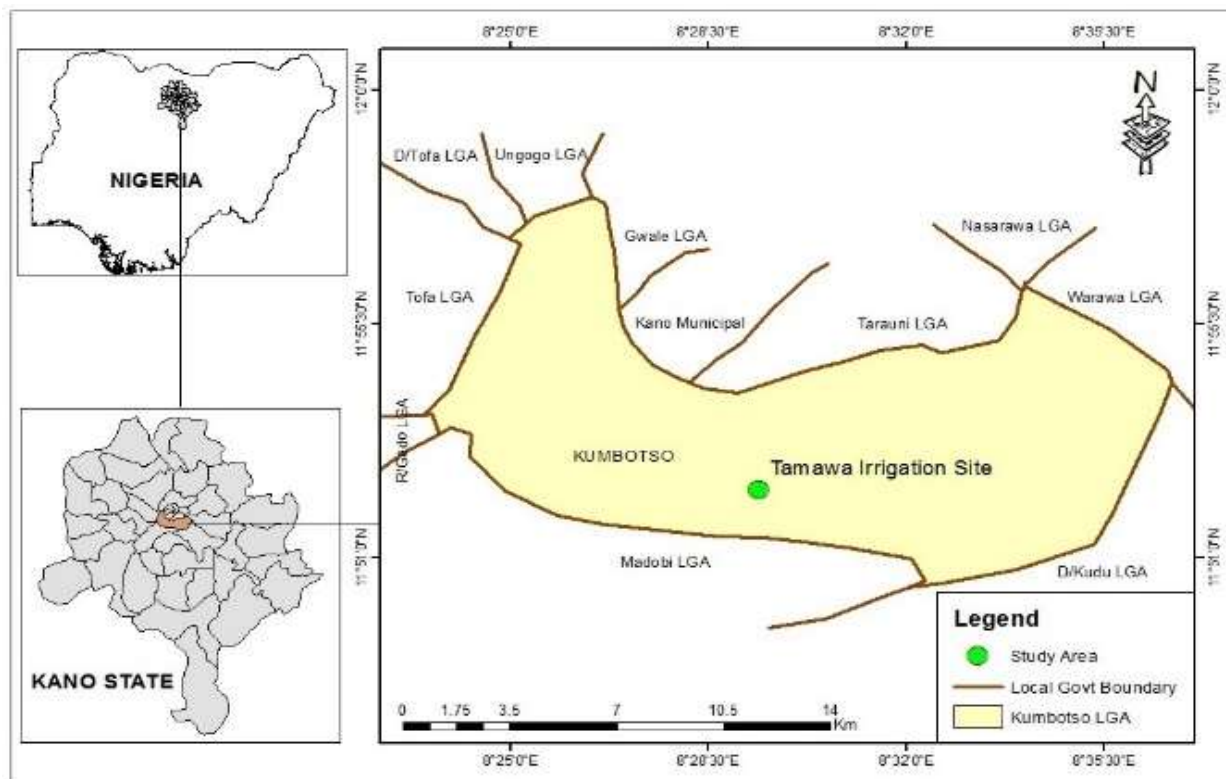
**Figure 2.** Map showing Kaita sample area in Katsina State, Nigeria



Kano state has more than 18,684 square kilometers cultivable (7,214 sq. mi) of land and is the most extensively irrigated state in the country. Kano has a tradition of irrigated agriculture and is reckoned as the leading hydro agricultural state in Nigeria [24] The sampling site at Tsamawa, Kumbotso local government area lies between latitude  $11^{\circ}50'S$  to  $12^{\circ}N$  and longitude  $8^{\circ}24'W$  to  $40'E$ . It falls within the Kano settlement Zone bordering the south and west by Madobi. Northwest by Rimingado and North by Gwale and East by Tarauni local government areas respectively. The regions soil is predominantly tropical ferruginous characterized by sandy textures with low water holding capacities [25], and the area is characterized by ancient crystalline rocks which comprises migmatite

gneiss, younger metasediments and granitic intrusions all of which had undergone metamorphic and tectonic activities [26].

Farmers obtain water for irrigation from a river that flows from Kusala at Karaye, to Madobi, to Challawa and then to Tsamawa. The irrigation area is in close proximity to many industries such as, Safari textile limited, Nigeria bottling company (Coca Cola), Fan milk industry, Mamuda agro-allied products, Fata tanning limited, etc. Sugarcane is the major plant grown there and other vegetables such as tomatoes, pepper, cucumber and onions.



**Figure 3.** Map showing Tsamawa sample area in Kano State, Nigeria

### **Sample collection and preparation**

The soil samples were collected at random from the irrigation site at Kofar Sauri, Kaita; and Tsamawa at uniform depth of 0-4 cm with the aid of hand trowel. The samples were collected in the month of November, 2023 in replicates which were mixed to form a homogenous sample. They were then air dried for several days at ambient temperature, pulverized and sieved through a 2mm stainless steel mesh [20]. A portion of the soil samples were properly labelled and taken to the soil laboratory at the department of Geography, Umaru Musa Yar'adua University, Katsina for physico-chemical parameters (pH, Cation exchange capacity (CEC), Water holding capacity (WHC), Organic carbon, (OC) Organic matter (OM) and Soil texture) analyses. Some of the prepared samples were measured, digested and taken to Central laboratory at BUK for heavy metals analyses using Atomic Absorption Spectrometry (AAS).

### **Digestion of soil samples**

To determine the heavy metals content in the soil samples wet digestion was carried out. The soil samples (1 g) were placed separately in a beaker with 10 ml mixture of HNO<sub>3</sub> and HCl in the ratio 3:1 (aqua regia). The beaker was heated to 100 °C on a hot plate until almost all the white fumes of nitrogen dioxide has evaporated. The digested soil

samples were then cooled and filtered through Whatman No. 4 filter paper. They were then transferred into graduated flasks and deionized water added up to the 50 ml mark [27]. The digests were then transferred to an acid-rinsed sample containers with a label for analysis.

The digests were analysed for their heavy metals (Cd, Cr, Cu, Mn, Ni, Fe, Pb and Zn) content in Central Laboratory, BUK, Kano using Atomic Absorption Spectrometer (AAS) Perkin Elmer, Pinacle 900H model.

### **Statistical analysis**

Data were reported as mean  $\pm$  standard deviation. Descriptive statistics were used in analyzing the data. Analysis of variance (ANOVA) was used to assess the significant differences between the mean concentrations of the heavy metals and also between the three investigated sites. Tukey's ANOVA was used to assess the significant difference between the adjusted probability values. Correlation matrix was used to determine the association between the levels of the investigated metals. and also, the investigated sites

## **RESULTS AND DISCUSSION**

Physico-chemical parameters analysis. The results obtained for physico-chemical parameters were presented in Table (1).

**Table 1.** Result of Physicochemical parameters of the soil samples from Kaita (KSS), Tsamawa (KISS) and Kofar Sauri (KUSS) irrigation sites.

Site	pH	WHC (%)	CEC (Cmol/Kg)	OC	OM	Sand (%)	Silt (%)	Clay (%)	Soil texture
KSS	6.10	75.53	6.43	2.43	4.20	32.4	15.4	52.2	Clay- loam
KISS	6.05	49.39	5.72	1.62	2.80	62.0	15.7	22.3	Sandy-clay- loam
KUSS	6.30	40..65	4.33	1.39	2.40	70.8	13.8	14.4	Sandy-loam

In Table (1) above the soil pH values ranged from 6.05 in KISS to 6.30 in KUSS indicating slightly acidic range at all the sampling units which is good for maintaining soil fertility and optimizing essential plant nutrients, as reported by [17] that slightly acidic soils support better bioavailability of essential nutrients, particularly phosphorus, and help limit the toxicity of certain heavy metals.[11] also indicated that acidic conditions may enhance metal mobility, increasing the potential for heavy metal uptake by plants. Other beneficial soil microorganisms also thrive better in slightly acidic conditions improving nutrient cycling. For instance, *Penicillium* was found to predominate at slightly acidic pH range [28].

The water holding capacity (WHC) and cation exchange capacity (CEC) ranged from 40.65 % (KUSS) to 75.53 % (KSS) and 4.33 Cmol/Kg (KUSS) to 6.43 Cmol/Kg (KSS) respectively. These two parameters greatly help in enhancing soil fertility. They both promote microbial activity by ensuring adequate moisture in the soil, retaining available nutrients and preventing leaching.

KSS has the highest WHC, likely due to its clay-loam texture, which retains more water compared to sandy soils. [8] observed that

high WHC enhances nutrient retention and plant growth, especially in semi-arid environments like Katsina and Kano. While KUSS has the lowest, correlating with its sandy-loam texture, which drains quickly and retains less water. [26] identified low WHC in sandy soils of the Kano region which limits their agricultural potential without supplemental irrigation.

CEC was found to be highest in KSS suggesting greater nutrient retention capacity and higher fertility. According to [12], CEC is enhanced by clay and organic matter, both abundant in KSS. While CEC in KUSS was lower which aligns with its sandy texture and lower organic content.[5] emphasized that soils with low CEC are more susceptible to nutrient leaching and metal mobilization, consistent with the sandy profile of KUSS

KSS has the highest value of organic carbon (OC) (2.43), followed by KISS (1.62), while KUSS has the least (1.39); for organic matter (OM) the trend is the same as in OC, the values are 4.20, 2.80 and 2.40 for KSS, KISS and KUSS respectively. [16] recorded total organic carbon in peri-urban farm soils to be within the range of 0.68- 6.32, suggesting possibility of metals retention within the soil, while Organic matter in the same soils ranged

from low to high with values varying between 1.18-10.8 which correspond with the findings of this study.

KSS has the highest value of OM which supports better microbial activity, nutrient cycling, and metal-binding capacity. [15] reported that organic matter helps in immobilizing heavy metals, thereby reducing their bioavailability. Whereby, low organic matter in KUSS reduces its capacity to buffer heavy metals and nutrients as reported by [6] that low organic matter soils, allow for higher metal mobility and potential plant uptake, increasing contamination risks. OC is a key component of OM, they increase CEC by providing sites for nutrient exchange [13].

The soil texture for KSS is clay- loam having 52.2 % clay, 32.4 % sand and 15.4 % silt; KISS is sandy- clay- loam having 62.0 % sand, 22.3 % clay and 15.7 % silt; while

KUSS is sandy- loam having 70.3 % sand, 14.4 % clay and 13.8 % silt.

KSS, with the highest percentage of clay, is likely to retain both water and metals better than the other two sites. This result agreed with the findings of [2] who highlighted that clay-rich soils can bind metals tightly, making them less bioavailable. KUSS on the other hand, has the highest percentage of sand making it the least effective at retaining both nutrients and metals, increasing leaching and contamination risks downstream. According to [14], sandy soils like those at KUSS can act as conduits for heavy metal mobility due to poor adsorption capacity.

Heavy metals analysis. The results for AAS analysis of the soil samples from the three irrigation sites were presented in Table (2).

**Table 2.** Mean heavy metal concentrations (mg/kg) in soils from Kofar Sauri (KUSS), Kaita (KSS) and Tsamawa (KISS) irrigation sites.

Heavy metals	KUSS	KSS	KISS	FAO/WHO
<b>Cd</b>	0.118 ± 0.001	1.443 ± 0.008	0.074 ± 0.001	0.01
<b>Cr</b>	2.948 ± 0.012	2.417 ± 0.008	2.060 ± 0.049	1.30
<b>Cu</b>	0.964 ± 0.003	3.882 ± 0.018	0.419 ± 0.002	10.00
<b>Fe</b>	14.56 ± 0.106	12.42 ± 0.024	14.12 ± 0.043	40.00
<b>Mn</b>	6.372 ± 0.020	3.422 ± 0.037	7.188 ± 0.037	0.08
<b>Ni</b>	1.173 ± 0.008	0.860 ± 0.011	0.789 ± 0.008	0.05
<b>Pb</b>	0.852 ± 0.018	1.749 ± 0.006	0.420 ± 0.017	0.10
<b>Zn</b>	1.701 ± 0.003	1.699 ± 0.005	0.919 ± 0.003	0.60

In table (2) above all the sites exceed FAO/WHO limits for Cd, Cr, Mn, Ni, Pb and Zn indicating potential heavy metal contamination. Kaita (KSS) soil shows the highest contamination for Cd, Cu and Pb with concentration values of 1.443mg/kg, 3.882mg/kg and 1.749mg/kg respectively. Kofar Sauri (KUSS) soil has the highest in

Cr, Ni and Zn with concentration values of 2.948mg/kg, 1.193mg/g and 1.701mg/kg respectively. KISS has the lowest concentrations in all the metals.

Cadmium (Cd) level exceeds safe limit (0.01 mg/kg) at all sites with highest concentration value of 1.443 mg/kg recorded in KSS. Cd

accumulation in agricultural soils over time is induced by human activities [29], such as excessive application of phosphate fertilizers and pesticides, industrial effluents and waste water from domestic discharges and contaminated underground water. [15] documented similar Cd contamination in tannery-affected regions, suggesting KSS may have anthropogenic sources such as contaminated underground water or agrochemical usage. Cd is highly toxic and associated with renal, bone, and reproductive disorders. [9] reported Cd as causing kidney damage and cancer.

Chromium (Cr) level in all the sites exceed the FAO/WHO limit of 1.30 mg/kg. Highest concentration was recorded in KUSS (2.948 mg/kg), a site receiving effluent from urban and industrial activities (e.g., tanneries, welding, painting). [5] and [11] observed that Cr accumulation often stems from industrial discharge. This report supports the idea that KUSS's urban proximity increases contamination risk.

Manganese (Mg) is a very essential trace metal for plants and animal's growth. Its deficiency produces severe skeletal and reproductive abnormalities in mammals. High concentration of Mn causes hazardous effects on lungs and brains of humans [3]. In this study Mn levels are critically high, especially in KISS with a value of 7.188 mg/kg, KUSS has 6.372 mg/kg, while KSS has the lowest value of 3.422 mg/kg, indicating concentrations higher than the standard limits of FAO/WHO in all the sites. [7] stated that Mn often accumulates from irrigation using low-quality water, a known issue in KISS and KUSS. The result from this study does not correspond with the findings of [30] where values ranging between 0.08 to

0.09 mg/kg were recorded in agricultural soils from Gonglung area, Jere.

Nickel (Ni) is carcinogenic and allergenic and it tends to persist in soils, increasing long-term exposure risks [5]. In this study all sites exceed the threshold limit set by FAOWHO (0.05 mg/kg); highest concentration (1.173mg/kg) was recorded in KUSS. [6] and [14] mentioned that Ni is mobile in sandy soils (like KUSS) which explained the reason behind highest concentration value of Ni in KUSS soil. These findings correspond with the reports by [28] who recorded higher concentrations values (0.37 g/kg – 0.06 g/kg) of Ni in irrigated urban area above USEPA maximum permissible limit which may be attributed to wastewater use. Nickel levels above recommended values can enter into vegetative biomass of vegetables and when consumed by humans can cause lung, liver and kidney damages [10]. It can also cause cancer, respiratory failure, birth defects, allergies, nervous system and heart failure [11].

Lead (Pb) is highly toxic, especially for children, causing cognitive impairment and neurological disorders. Inhalation and ingestion of Pb instigates long term harm in adults and pregnant women with increased risk of high blood pressure and birth deformities respectively [31, 9] emphasized Pb's link to neurodevelopmental issues. Owing to its relevance and usefulness in the metallurgical industry, Highest mean concentration of Pb were recorded in this study which are far above the maximum permissible limit set by FAO/WHO (0.10 mg/kg). KSS has the highest Pb concentration (1.749 mg/kg) which might be as a result of its geological sources, and the

lowest concentration was found in KISS (0.852 mg/kg). [15] stated that Pb presence in irrigation systems can reduce microbial diversity affecting soil health.

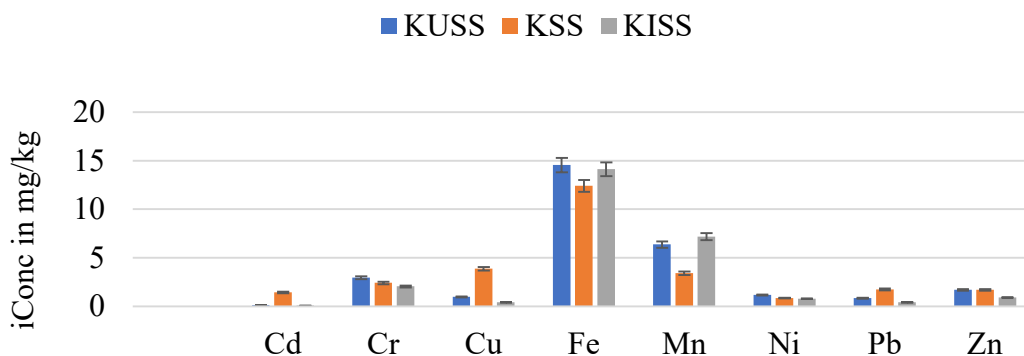
Copper (Cu) concentrations in all the sites are below the 10 mg/kg limit, hence not posing direct toxicity risks. KSS has the highest Cu (3.882 mg/kg), possibly from natural geological sources (like the Gundumi formation) or use of Cu-based agrochemicals. This finding coincides with the report from [4] which highlighted that Cu from fertilizers and wastewater can accumulate in clay-rich soils like KSS [2] also noted Cu's dual role as both a micronutrient and a pollutant. [8] identified Cu spikes in wastewater-irrigated soils but well below the toxic threshold, similar to KSS findings.

Iron (Fe) levels in all sites are well below the WHO limit of 40mg/kg with KSS having the lowest concentration (12.42 mg/kg), while 14.56 mg/kg and 14.12 mg/kg were recorded in KUSS and KISS respectively. Though not considered toxic at these levels, high Fe can alter soil chemistry and interact with other metals, affecting plant nutrient uptake [12]. [13] emphasized Fe's role in modifying

mobility of Pb and Zn under varying redox conditions. Stable Fe levels across the sites reflect background geogenic sources, not pollution.

Zinc (Zn) though essential, is toxic at high concentrations it inhibits photosynthesis and enzyme activity. In this study concentration values of 0.919 mg/kg (KISS) – 1.701 mg/kg (KUSS) were recorded which are all well above the safe limit (0.60 mg/kg), this might be associated to irrigation water sources (Tube wells, urban discharges and industrial discharges) in the sites. This does not correspond with the findings of [20] which obtain Zn values below standard limits in irrigation soil from Katsina urban irrigation site Zn mobility is influenced by pH and organic matter, which explains variation across sites [28].

Table 3 – 4 below represents statistical analyses of data obtained to determine significant difference between heavy metal concentrations and between irrigation sites using Analyses of variance (ANOVA), also between the adjusted probability values (Tukey's ANOVA)).



**Figure 4.** Graphical representation of Heavy metals concentrations in KUSS, KSS and KISS

**Table 3.** ANOVA for heavy metal concentrations and irrigation sites

Sources	Df	Sum sq	Mean sq	F-value	Pr(>F)
<b>MC</b>	7	1238.5	176.93	6.645	0.000324
<b>IGS</b>	3	57.3	19.10	0.717	0.5527
<b>RESIDUALS</b>	21	559.2	26.63		

From the results obtained in Table (3) it is observed that the probability value for irrigation sites (IGS) is (0.5527) which is greater than the alpha level (0.05), thus, we fail to reject the null hypothesis and conclude that there is no significant difference between the means of the irrigation sites. Despite the data presented in Table (2) showing KSS with the highest concentrations of Cd and Pb, and KUSS with the highest concentrations of Cr, Ni and Zn, the overall variation between sites is statistically insignificant. This Suggests widespread and homogeneous contamination potentially from common practices like: untreated wastewater use [18], over-application of agrochemicals and background geological enrichment, especially in KSS with Gundumi formations [22, 8] found that wastewater irrigation along Katsina's Ginzo channel contributed uniformly to soil pollution, which aligns with this study's results. [20] reported no significant site-based variation in metal concentrations across Katsina's urban farms matching this ANOVA finding.

The probability value for metal concentrations (MC) is (0.000324) which is less than the alpha value, therefore we are to reject the null hypothesis and conclude that

there is significant difference between the means of the metal concentrations. This confirms findings from table 2, where Metals like Cd, Cr, Mn, Ni, Pb, and Zn exceeded FAO/WHO safe limits. While Fe and Cu remained within acceptable thresholds. Certain metals are more dominant pollutants and thus pose higher environmental and health risks. This supports a metal-specific remediation strategy as stipulated by [2] and [12] who emphasized the need to differentiate between toxic and essential metals in soil risk assessments.

The residual variance represents unexplained differences in the data. This indicates potential influence from factors not accounted for, such as: sampling time, microbial activity, soil mineralogy, depth variability and seasonal irrigation practices. According to [17], spatial variability in metal uptake and retention in soils is influenced by microclimate, organic matter quality, and soil structure, which may contribute to residual variance.

**Table 4.** ANOVA for Adjusted Probability Values

MC	Mean Difference	Lower bound	Upper bound	Adjusted p – value
Fe-Cr	18.09375	5.855257	30.332243	0.0014202
Fe-Cu	16.45875	4.220257	28.697243	0.0039364
Mn-Fe	-16.00950	-28.247993	-3.771007	0.0052033
Ni-Fe	-19.55700	-31.795493	-7.318507	0.0005713
Pb-Fe	-19.49475	-31.733243	-7.256257	0.0005937
Zn-Fe	-19.04525	-31.283743	-6.806757	0.0007850

This test was conducted post-ANOVA to determine which specific metal concentrations differ significantly from others. The comparison is made between Fe and other metals (Cr, Cu, Mn, Ni, Pb, Zn). From the results obtained in Table 4 it is observed that all the adjusted probability values are less than 0.05, therefore, this confirmed that there exists a significant difference between the observed metal concentrations (i.e. each pair compared with Fe is significantly different in concentration). The confidence intervals (Lower–Upper) do not include zero, strengthening the reliability of these differences.

Fe (14.56–14.12 mg/kg) is within safe FAO/WHO limits (40 mg/kg) and less variable across sites. Metals like Mn (up to

7.188 mg/kg vs 0.08 FAO/WHO limit), Ni (1.173 vs 0.05), and Pb (1.749 vs 0.10) far exceed FAO/WHO limits, explaining why their concentrations are statistically distinct from Fe [9] and [15]. highlight Pb, Cd, Mn, and Ni as priority pollutants due to their high toxicity and tendency to persist in agricultural systems. [28] shows Zn often accumulates in heavily irrigated soils and can cause toxicity in plants at high levels, consistent with Zn’s significant difference from Fe.

Table 5 and 6 represents correlation matrixes for the heavy metals and the irrigation sites to determine possible association between the heavy metals and the irrigation sites.

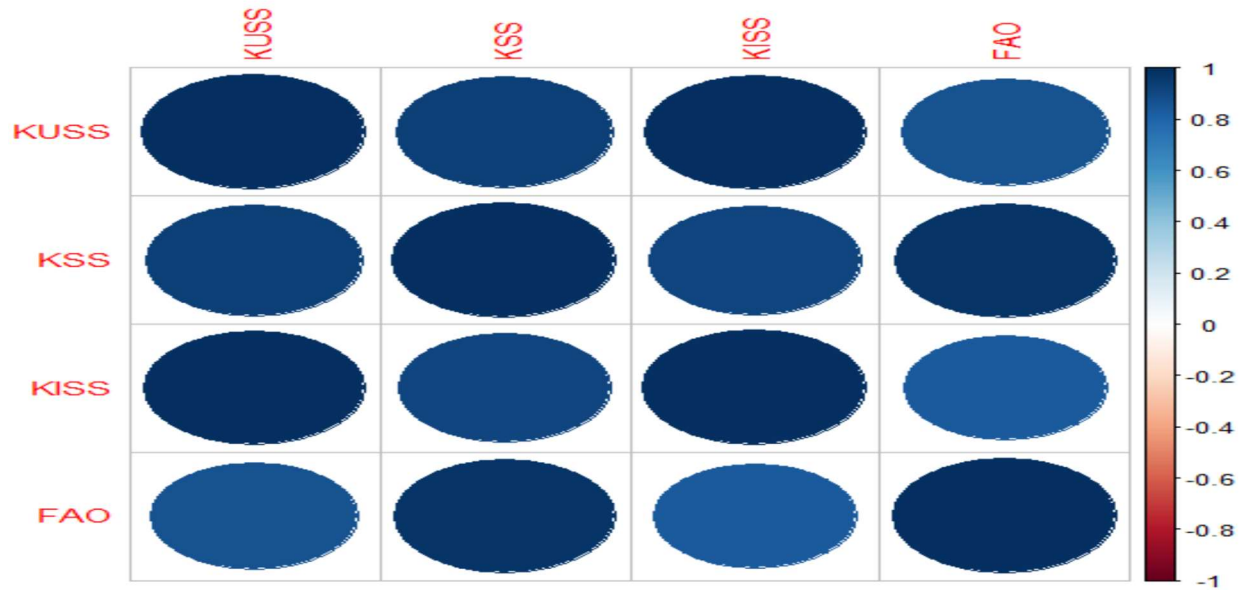
**Table 5.** Correlation Matrix for Irrigation Sites

Variable	KUSS	KSS	KISS	FAO
<b>KUSS</b>	1.0000000	0.9361225	0.9944231	0.8677178
<b>KSS</b>	0.9361225	1.0000000	0.9132431	0.9791853
<b>KISS</b>	0.9944231	0.9132431	1.0000000	0.8319281
<b>FAO</b>	0.8677178	0.9791853	0.8319281	

This matrix (table 5) evaluates the degree of linear relationship among the three irrigation sites, KUSS (Kofar Sauri), KSS (Kaita), and KISS (Tsamawa) along with FAO/WHO reference values. Correlation values (r) range

from -1 to +1 (+1 = perfect positive correlation, 0 = no correlation and -1 = perfect negative correlation).





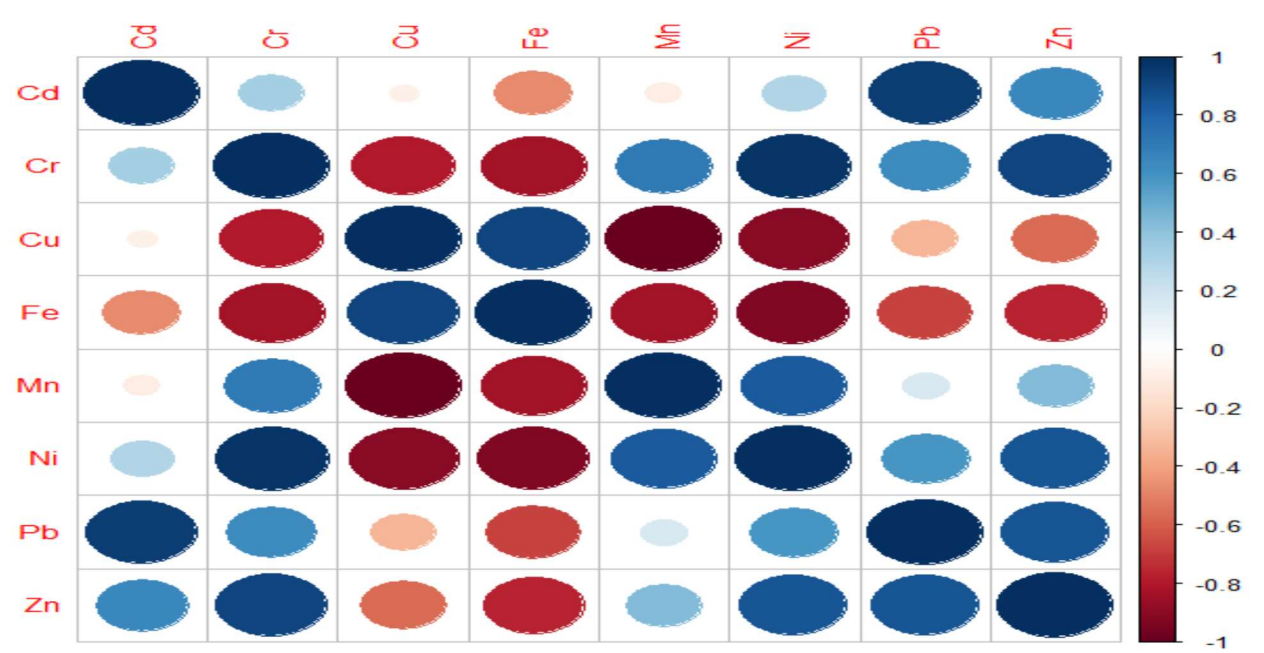
**Figure 5.** Correlation Plot for Irrigation Sites

From the results obtained in Table 5 and Figure 5 it is observed that there is strong positive correlation between the irrigation sites. The very high correlation (especially between KUSS and KISS) suggests that these sites may be exposed to common pollution sources such as: wastewater irrigation, atmospheric deposition and agrochemical runoff. They may also share soil types or drainage basins, especially in the case of KUSS (urban, waste-channel-fed) and KISS (linked to Kano's industrial river). KSS which is somewhat geologically distinct due to its Gundumi formation, still shows high correlation (0.91–0.94), implying that widespread regional contamination exists.

[20] and [8] reported consistently high heavy metal levels across urban irrigation sites in Katsina, regardless of location confirming this matrix's outcome. [7] emphasized that shared land use patterns (like farming near settlements), and shared water sources (like rivers and drainage basins), lead to correlated pollution patterns across different locations. [17] found that even geologically distinct areas can show homogenized contamination due to long-term anthropogenic impacts aligning with the high correlation of KSS to others.

**Table 6.** Correlation Matrix for Heavy Metals

Variable	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Zn
Cd	1.0000	0.3306	-0.0704	-0.4709	-0.0983	0.2978	0.9437	0.6463
Cr	0.3306	1.0000	-0.7908	-0.8362	0.7099	0.9754	0.6241	0.9189
Cu	-0.0704	-0.7908	1.0000	0.9131	-0.9849	-0.9030	-0.3312	-0.5641
Fe	-0.4709	-0.8362	0.9131	1.0000	-0.8307	-0.9214	-0.6792	-0.7648
Mn	-0.0983	0.7099	-0.9849	-0.8307	1.0000	0.8350	0.1630	0.4302
Ni	0.2978	0.9755	-0.9030	-0.9214	0.8350	1.0000	0.5868	0.8532
Pb	0.9437	0.6241	-0.3312	-0.6792	0.1630	0.5868	1.0000	0.8582
Zn	0.6463	0.9189	-0.5641	-0.7648	0.4302	0.8532	0.8582	1.0000

**Figure 6.** Correlation Plot for Heavy Metals

The matrix presented in Table 6 shows the Pearson correlation coefficients ( $r$ ) between the concentrations of different heavy metals (Cd, Cr, Cu, Fe, Mn, Ni, Pb, Zn) in the soil samples across all three irrigation sites.

From the results obtained in Table 6 and Figure 6 it is indicated that there are strong positive ( $r > 0.8$ ) correlation which might be as a result of common or overlapping pollution sources as found between Cd–Pb, Cr–Ni, Cr–Zn, Fe–Mn, etc. [15], reported strong co-mobilization of Cd and Pb in soils irrigated with industrial wastewater, confirming the Cd–Pb correlation. In another report by [2], Ni, Cr, and Zn often co-occur in soils exposed to tanneries, electroplating, and sewage discharge, matching the Cr–Ni–Zn pattern here. Moderate positive correlation exists between Cd–Zn and weak positive correlation also between Cd–Ni.

Strong negative correlation ( $r < -0.8$ ) which might be as a result of biogeochemical interactions, potentially influenced by soil pH, texture, or microbial activity was found between Cr–Cu, Cr–Fe and Fe–Ni. [5] found that metal interactions can be antagonistic or synergistic depending on pH, organic matter, and redox state, explaining Cu–Mn, Cu–Ni negative relationships ( $r > -0.8$ ). Moderate negative correlation exists between Cd–Fe and weak negative correlation was found between Cd–Cu in the irrigation sites. Mostly heavy metals such as Cd, Pb, Ni, Cr, Zn are interrelated, pointing to combined and systemic contamination. Strong correlations reveal metal clusters, which should be jointly monitored and remediated while, antagonistic relationships may influence metal uptake by crops, necessitating plant-metal interaction studies

## CONCLUSION

Kaita (KSS) soil exhibits superior physico-chemical qualities (high WHC, CEC, OC, and a favorable clay-loam texture) ideal for agriculture. However, it also had the highest heavy metal contamination, suggesting input from external pollution sources such as the groundwater used for irrigation, mobilization from Gundumi formations or the soil itself that was formed from weathering of rocks. Tsamawa (KISS) and Kofar Sauri (KUSS) had less favorable soil characteristics, especially KUSS, which has low CEC and OM and a sandy texture, compounding risks of nutrient and metal leaching. These findings suggested that soil quality alone cannot mitigate heavy metal risks; site-specific pollution inputs are crucial in determining contamination levels. Analyses of variance conducted showed significant difference in mean metal concentrations and no significant difference in the irrigation sites. Tukey's ANOVA also confirmed significant variation in metal concentrations. Correlation matrix analyses also showed existence of strong positive correlation between the irrigation sites and variable correlation (strong positive, moderately positive, weak positive, strong negative, moderately negative and weak negative correlations) between the metal concentrations. According to [27], soils contaminated with Pb, Cd, or Cr require urgent intervention due to risks of crop contamination, bioaccumulation, and human toxicity. This suggested potential health and environmental risks, requiring remediation strategies and further investigation into the contamination sources for control such as,

industrial effluent regulation and wastewater treatment. Routine monitoring and crop-metal uptake studies are needed to prevent food-chain transfer.

### CONFLICT OF INTEREST

Authors declared no conflict of interest.

### FUNDING

There was no funding received for this work.

### ETHICAL STATEMENT

This work required no ethical statement.

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# Proximate, Physicochemical, Elemental, and Amino Acid Profile of *Fragaria x ananassa* (Strawberry) Grown on the Plateau

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Received: 4<sup>th</sup> May, 2025

Revised: 24<sup>th</sup> June, 2025

Accepted: 18<sup>th</sup> August, 2025

## Abstract

This study evaluated the proximate, phytochemical, elemental, and amino acid composition of *Fragaria x ananassa* fruit. Proximate analysis revealed high carbohydrate (33.43%) and ash (28.48%) contents, alongside moderate crude fiber (21.26%) and low protein (3.24%) and fat (1.50%) levels. Qualitative and quantitative phytochemical screenings indicated high concentrations of phenols, saponins, and sugars, with ethanol extracts yielding greater amounts than aqueous extracts. Elemental analysis showed magnesium (18.99 mg/kg) and calcium (14.01 mg/kg) as the most abundant essential metals. However, chromium (0.31 mg/kg) and platinum (0.41 mg/kg) levels slightly exceeded WHO/FAO permissible limits, suggesting potential health concerns. Amino acid profiling identified aspartic acid (0.1493 mg/ml) and glutamic acid (0.0952 mg/ml) as predominant.

**Keywords:** Strawberry, *Fragaria x ananassa*, amino acid profile, proximate, phytochemical, elemental analyses.

## INTRODUCTION

Strawberries *Fragaria x ananassa* are globally cherished fruits recognized for their unique flavor, attractive red color, and rich nutritional profile [1]. Traditionally, various parts of the strawberry plant—including the fruit, leaves, and roots—have been used in ethnomedicine for treating ailments such as gastrointestinal disorders, liver complaints, skin conditions, and as diuretics or mild laxatives [2,3]. The fruit, in particular, is widely used in herbal teas to relieve diarrhea, support kidney function, and reduce inflammation [4].

Strawberries are notable sources of vitamin C, which contributes significantly to diabetes management [5], enhanced immune function [6], and may retard leukemia development [7]. Among red berries, strawberries particularly stand out due to their qualitative and quantitative characteristics [8]. Widely consumed [9], their nutrient content varies with cultivation systems [10], maturation stages [11], climate conditions [12], and post-harvest practices [13-14].

The primary attributes influencing strawberry acceptability include their red color, sweet taste, and juicy texture [15],

with color mainly resulting from anthocyanin accumulation during maturation [11]. Nutritionally, strawberries surpass fruits like citrus, guava, and apple in protein, mineral, and vitamin content [8, 16, 17]. In Nigeria, particularly in Plateau State, strawberries hold economic importance. They are highly valued not only for sensory qualities but also for their phytochemical content [18], offering antioxidant, anticarcinogenic [19], antimutagenic [20], anti-inflammatory [21], and antihypertensive benefits [22].

Despite their cultivation, gaps persist in understanding the nutritional properties of local Nigerian strawberries, with challenges linked to pest management, post-harvest losses, storage methods, and nutritional profiling. Strawberries are particularly perishable, with shelf-life of 1-2 days at room temperature, necessitating prompt consumption or processing [23]. Effective temperature management remains vital to extending post-harvest life. Genetically, strawberries are a hybrid species. The cultivated garden strawberry, *Fragaria × ananassa*, popularly known for low-growing habits and aggregate fruits with embedded seeds, is an octoploid hybrid that originated approximately 250 years ago through a natural cross between the North American



species *F. virginiana* and the South American species *F. chiloensis* [8].

Strawberries are processed into various products like jams, yogurts, and cosmetics, leveraging their abundant nutrients, including vitamins (C, E, K, B-complex) and minerals like iron, calcium, and potassium [24-27]. Additionally, their bioactive compounds—phenolic acids, flavonoids, tannins, alkaloids, and terpenoids—possess antioxidant, anti-inflammatory, antimicrobial, and anticancer properties [28-32]. Understanding these attributes is essential for maximizing strawberries' health and economic potential.

Recent studies emphasize strawberries' enhanced bioactive content under organic farming conditions [33], improved antioxidant stability with cold storage innovations [34], and promising applications in functional foods and pharmaceuticals [35]. Advances in metabolomics profiling reveal key compounds responsible for strawberries' therapeutic properties [36], while novel breeding techniques target enhancing their shelf-life and nutritional quality [37]. These developments underline the growing significance of strawberries in health promotion and economic sustainability.

## **MATERIALS AND METHODS**

### **Collection of Plant Material and Extraction**

Fresh strawberry fruits (*Fragaria* × *ananassa*) were collected from Chaha, Vom, located in Jos South Local Government Area (LGA) of Plateau State, Nigeria. The fruits were thoroughly washed with clean water, air-dried at room temperature, and subsequently ground into a fine powder using a mortar and pestle. A total of 307 g of the powdered fruit was weighed and stored in a sterile, dry bag for subsequent analyses.

### **Extraction and Concentration**

For the extraction, 150 g of the finely powdered strawberry (*Fragaria* × *ananassa*) sample was subjected to Soxhlet extraction using 500 mL of a 70:30 ethanol-to-water (v/v) solvent mixture for 24 hours. The extraction was carried out at an ideal temperature range of 60–70 °C, consistent with the recommendations of the Association of Official Analytical Chemists (AOAC, 2019), to prevent the degradation of thermolabile compounds. This hydroethanolic solvent system was chosen for its ability to extract both moderately polar and highly polar compounds, including phenols, flavonoids, tannins, saponins,

terpenes, anthraquinones, alkaloids, coumarins, and reducing sugars [38].

Ethanol enhances the solubility of moderately polar phytochemicals such as flavonoids and terpenes, while water facilitates the extraction of polar compounds like anthocyanins, phenolic acids, sugars, and some amino acids. This dual-polarity ensures efficient recovery of antioxidant-rich bioactive compounds while preserving their structural integrity [39].

Additionally, the extraction aimed to isolate amino acids such as tryptophan, threonine, isoleucine, leucine, lysine, methionine, glutamic acid, phenylalanine, tyrosine, valine, arginine, histidine, alanine, aspartic acid, cysteine, glycine, proline, and serine, which are biologically essential and often co-extracted with polyphenols in aqueous-alcoholic solvents [40]. These amino acids contribute to the nutritional and therapeutic relevance of strawberries.

The resulting extracts were concentrated using a hot water bath at temperatures below 50 °C to retain bioactivity and stored at 4 °C for subsequent qualitative and quantitative analyses.

### Proximate Analysis

All the proximate analysis of moisture content, ash content, crude fat, crude fibre, crude protein, and carbohydrate of *Fragaria* × *ananassa* were determined using standard analytical method according to (AOAC 2005).

### Determination of Moisture Content

An aluminum moisture pan was washed, dried in an oven, cooled in a desiccator, and weighed. Approximately 100 g of the sample was placed in the pan and weighed. The pan with the sample was then oven-dried at a temperature range of 80–100 °C, checked at 3-hour intervals, until a constant weight was achieved. After cooling, the final weight was recorded, and the moisture content (%) was calculated. Drying within this temperature range, rather than at a fixed 100 °C, was necessary due to the highly perishable nature of strawberries, which typically have a shelf life of only 1–2 days at room temperature. Using a moderate drying range allows for gradual moisture removal while minimizing structural or chemical alterations.

After cooling, the final weight was recorded. Moisture content (%) was calculated as:

$$\frac{(W_2) - (W_3)}{(W_2)} \times 100$$

Where,

Weight of pan + wet sample ( $W_2$ )

Weight of pan and dry sample ( $W_3$ )

Weight of empty pan ( $W_1$ )

### Determination of Ash Content

A clean crucible was dried in a muffle furnace, cooled in a desiccator, and weighed. About 10 g of the *Fragaria* × *ananassa* sample was added and reweighed. The crucible and sample were gradually heated from 200 °C to 450 °C in a muffle furnace for 4–5 hours to obtain ash. This temperature range was used to prevent charring and spattering due to the fruit's high sugar and moisture content. After cooling in a desiccator, the final weight was recorded.

Ash content (%) was calculated as:

$$\frac{(W_3) - (W_1)}{(W_2) - (W_1)} \times 100$$

Weight of crucible + sample ( $W_2$ )

Weight of crucible + ash ( $W_3$ )

Weight of empty crucible ( $W_1$ )

### Determination of Fibre Content

10 g of the sample was weighed into a 50 mL conical flask, and 100 mL of 1.25% sulfuric acid ( $H_2SO_4$ ) was added. The mixture was gently boiled for 30 minutes

under reflux and filtered through a poplin cloth using a Buchner funnel. The residue was washed with hot distilled water and then treated with 100 mL of 1.25% sodium hydroxide (NaOH), 2–3 mL of distilled water, and 3–5 drops of vegetable oil as an anti-foaming agent. It was again gently boiled for 30 minutes, filtered, and thoroughly rinsed with hot distilled water, 10–15 mL of 95% ethanol, and 10–15 mL of ether. The residue was oven-dried at 105 °C for 2 hours, cooled in a desiccator, and weighed. It was then ashed in a muffle furnace at 300 °C for 30 minutes, cooled, and reweighed. Crude fibre (%) was calculated as. Crude fibre (%) was calculated as:

$$\frac{(W_2) - (W_3)}{(W_2)} \times 100$$

Where,

Weight of residue + crucible ( $W_2$ )

Weight of sample from the muffle furnace ( $W_3$ )

Weight of sample ( $W_1$ )

### Determination of Crude Fat Content

A weighed thimble was filled with about 10 g of the sample and weighed again. A 500 mL round-bottom flask was also weighed. The Soxhlet extractor, fitted with a reflux

condenser, was used with petroleum ether as solvent. The system was heated to allow extraction for 5–6 hours. Afterward, the solvent was evaporated, and the residue was oven-dried at 100 °C, cooled in a desiccator, and weighed. The % extracted lipid is given by the formula. Crude fat (%) was calculated as:

Weight of the residue ( $W_4$ )

Weight of round bottom flask ( $W_3$ )

Weight of the sample ( $W_2$ )

Weight of the thimble ( $W_1$ )

### Determination of Protein Content

The crude protein content was determined by the Kjeldahl method involving digestion, distillation, and titration:

**Digestion:** 0.5 g of the sample was mixed with 2 mL HCl, 3 mL  $\text{HNO}_3$ , 45 mL distilled water, and 1 g mercury oxide (catalyst). The mixture was digested on a hot plate under a fume cupboard at 250 °C for 30–40 minutes.

**Distillation:** The digest was distilled using a Kjeldahl apparatus and diluted to 100 mL with distilled water.

**Titration:** The distillate was titrated against 0.02 N HCl.

The percentage nitrogen and protein were calculated as:

Nitrogen free extract

$$\frac{[(V_1 - V_2)(14.01 \times N)]}{W \times 1000} \times 100$$

%NFE =

Where:

N = Normality of acid, W = Weight of the sample,  $V_1$  = Titre value,  $V_2$  = Blank value, %protein = % Nitrogen free extract x conversion factor.

Conversion factor, 6.25 was used

### Determination of Carbohydrate Content

The carbohydrate can be represented by the stoichiometric formula ( $C_nH_2O$ ) where n is the number of carbon in the molecules. Therefore, the ratio of carbon to hydrogen to oxygen is 1:2:1 in the carbohydrate molecules. The total carbohydrate was determined by difference in method in which the sum of the % of moisture, ash, crude fibre, crude fat and protein content were subtracted from the sample. That is, (100 - % moisture content, ash content, crude fiber content, crude fat content and protein content). % Carbohydrate = 100 - (% Moisture + % Ash + % Crude Fibre + % Fat + % Protein)

### Qualitative Phytochemical Analysis

The extract was screened for the presence of major phytochemicals following the AOAC Official Methods of Analysis (2019 edition) [41].

The tests conducted include phenols, flavonoids, tannins, saponins, terpenes, anthraquinones, alkaloids, coumarins, and reducing sugars.

Each test was performed in triplicates and interpreted based on specific observable colour changes or precipitate formation.

**Test for Phenols:** To 1 mL of the extract, 2 mL of distilled water and a few drops of 10% ferric chloride were added. The formation of a bluish-green or dark blue colour indicated the presence of phenolic compounds.

**Test for Flavonoids (Sodium Hydroxide Test):** 1 mL of 10% sodium hydroxide solution was added to 1 mL of the extract. The appearance of a bright yellow colouration, which turned colourless upon the addition of dilute hydrochloric acid, confirmed the presence of flavonoids.

**Test for Tannins:** 1 mL of 5% ferric chloride solution was added to 1 mL of the extract. A blue-black or greenish-black precipitate indicated the presence of tannins.

**Test for Saponins (Froth Test):** 2 mL of the extract was mixed with 2 mL of distilled water and shaken vigorously in a test tube. Formation of a stable, persistent froth layer

of at least 1 cm height after 10 minutes indicated the presence of saponins.

**Test for Terpenes (Salkowski Test):** To 2 mL of the extract, 2 mL of chloroform was added, followed by 2 mL of concentrated sulfuric acid (carefully poured down the side). The formation of a reddish-brown interface confirmed the presence of terpenes.

**Test for Anthraquinones (Borntrager's Test):** 1 mL of the extract was boiled with 0.02 mL of 5% dilute sulfuric acid, cooled, and filtered. The filtrate was shaken with 5 mL of chloroform. The chloroform layer was separated and shaken with 1 mL of 10% ammonia solution. A pink to red colouration in the ammoniacal layer indicated the presence of free anthraquinones.

**Test for Alkaloids (Mayer's Test):** 2 mL of the extract was acidified with 1 mL of 5% dilute hydrochloric acid, followed by the addition of a few drops of Mayer's reagent. The formation of a creamy white or greenish precipitate confirmed the presence of alkaloids.

**Test for Coumarins:** 2 mL of the extract was mixed with 2 mL of 2% sodium hydroxide solution and heated in a boiling water bath for 3 minutes. Four drops of concentrated hydrochloric acid were then added. The

appearance of a cloudy white precipitate or fluorescence under UV light indicated the presence of coumarins.

Test for Reducing Sugars (Benedict's test): 1 mL of the extract was treated with 2 mL of Benedict's reagent and heated in a boiling water bath for 5 minutes. A brick-red, yellow, or green precipitate indicated the presence of reducing sugars.

### Quantitative Analysis Method

The extract (10 g) was weighed and dissolved in 1.0 ml of distilled water. The solution was transferred to centrifuge for 10 minutes. 1.0 ml of the upper layer was measured and transferred to the spectrophotometer. To read the absorbance at 436 nm, distilled water was used as blank. Calculating at absorbance 436 nm, =

$$\frac{A \times V \times DF}{\epsilon \times L \times W}$$

Where, A = Absorbance, mV = Volume of extract, DF = Dilution factor,  $\epsilon$  = Molar absorptivity, L = Path lens, W = Sample weight.

### Elemental Analysis

Fifty gram of dried sample was placed in a crucible and heated in a muffle furnace, gradually raising the temperature from 100 °C to 250 °C for 1–2 hours. After cooling in a desiccator, 20 g of the ash was

digested by adding 20 mL HNO<sub>3</sub>, 10 mL HCl, and 10 mL distilled water. The mixture was heated on a hot plate under a fume hood for 30–40 minutes, cooled, and diluted with 50 mL distilled water. It was then filtered twice using Whatman No. 1 filter paper. The filtrate was analyzed using Atomic Absorption Spectrophotometry (A.A.S) with element-specific hollow cathode lamps and adjusted wavelengths to detect essential and heavy metals.

### Amino Acid Content

A 100 g sample of the extract was placed in a round-bottomed flask, and 6 mL of sulfuric acid was added. The flask was connected to a reflux apparatus and gently heated for 24 hours to hydrolyze peptide bonds. After cooling in a desiccator, the aqueous layer containing amino acids was separated and drained into a clean container. The amino acids were then analyzed using an amino acid analyzer.

## RESULTS AND DISCUSSION

The proximate composition of *Fragaria × ananassa* fruit powder (Table 1) reveals a high carbohydrate (33.43 ± 1.63%) and crude fibre content (21.26 ± 0.15%), suggesting its significant contribution to energy provision and digestive health. This finding agrees with Zhang *et al.* [42], who reported that fruits like strawberries offer

substantial carbohydrates and fibre beneficial for maintaining gut health. The low fat content ( $1.50 \pm 0.17\%$ ) and moderate protein level ( $3.24 \pm 0.14\%$ ) further support its value for low-fat, balanced diets, aligning with observations by de Souza *et al.* [43] regarding the nutritional properties of fruits.

Phytochemical screening (Table 2) indicated the presence of phenols, flavonoids, saponins, and tannins in both ethanolic and aqueous extract, with ethanolic extraction yielding higher quantities (Table 3). The superior extraction efficiency of ethanol corresponds to the findings of Bhattacharjee *et al.* [44], who emphasized the effectiveness of organic solvents in retrieving bioactive compounds. Notably, high concentrations of phenols ( $6.01 \pm 0.01$  mg) and saponins ( $5.70 \pm 0.02$  mg) were recorded in the ethanolic extract, which implies a strong antioxidant potential, consistent with Shahidi and Ambigaipalan [45] and Khan *et al.* [46], who highlighted the health benefits of phenolic and saponin-rich foods. established by WHO [51]. However, chromium ( $0.31 \pm 0.01$  mg/kg) slightly exceeded the recommended threshold (0.25 mg/kg), raising concerns about environmental contamination during

Flavonoids and tannins, also detected at appreciable levels, are well known for their anti-inflammatory, cardioprotective, and antimicrobial activities [47, 48].

The elemental composition analysis (Table 4) showed magnesium ( $18.99 \pm 0.02$  mg/kg) and calcium ( $14.01 \pm 0.01$  mg/kg) as the dominant minerals. Magnesium's abundance is critical for enzymatic reactions, muscle function, and metabolic regulation, in agreement with Rosanoff *et al.* [49]. Calcium is similarly vital for bone health and metabolic functions [50]. Minor elements such as iron ( $0.55 \pm 0.01$  mg/kg) and sodium ( $1.50 \pm 0.00$  mg/kg) were also detected but in lower concentrations, indicating that while strawberries contribute to micronutrient intake, they may need to be complemented with other dietary sources to meet daily requirements.

Heavy metal analysis (Table 5) revealed very low concentrations of cadmium ( $0.02 \pm 0.01$  mg/kg) and lead ( $0.01 \pm 0.00$  mg/kg), both within safe limits (0.25 mg/kg), raising concerns about environmental contamination during cultivation. This result highlights the ongoing need for environmental monitoring

in agricultural practices, as stressed by Gupta and Gupta [52].

Amino acid profiling (Table 6) showed that aspartic acid ( $0.1493 \pm 0.0002$  mg/ml) and glutamic acid ( $0.0952 \pm 0.0003$  mg/ml) were predominant, with essential amino acids like leucine, lysine, and threonine also present.

These amino acids play pivotal roles in protein synthesis, neurotransmitter functions, and overall metabolic health [53]. The presence of essential and non-essential amino acids enhances the nutritional significance of *F. × ananassa*, corroborating the functional food potential described by Kaur and Kapoor [54].

**Table 1.** Result for Proximate Composition of *Fragaria x ananassa* fruit

Parameter	Percentage value (%)
Moisture content	12.09±0.60
Ash content	28.48±0.54
Crude fibre content	21.26±0.15
Crude fat	1.50±0.17
Crude protein	3.24±0.14
Carbohydrate	33.43±1.63

*Value reported as Mean ± SD for triplicate analysis n = 3*



**Table 2.** Phytochemical Screening Result for Fruit of *Fragaria x ananassa*

Constituent	Extract with ethanol	Extract with distilled water
Phenols	+++	+++
lavonoids	++	++
Tannins	++	+
Saponins	+++	++
Terpenes	++	+
Anthraquinones	+	-
Alkaloids	++	+
Coumarins	++	++
Sugar	+++	+++

**Keys:** +++ = *High concentration*, ++ = *Moderate concentration* + = *Low concentration*, - = *Not detected*

**Table 3.** Quantitative Phytochemical Screening Test Result for fruit of *Fragaria x ananassa*

Constituent	Extract with ethanol (mg)	Extract with distilled water (mg)
Phenols	6.01±0.01	4.50±0.01
Flavonoids	3.20±0.01	2.50±0.00
Tannins	3.50±0.02	1.90±0.01
Saponins	5.70±0.02	2.80±0.01
Terpenes	3.00±0.01	1.60±0.01
Anthraquinone	1.40±0.02	0.05±0.02
Alkaloid	3.90±0.02	2.00±0.01
Coumarins	3.00±0.00	2.53±0.01
Sugar	5.03±0.03	4.98±0.03

*Value reported as Mean ± SD for triplicate analysis n = 3*

**Table 4.** Result for Essential metal concentration of *Fragaria x ananassa*

Elements	Concentration (mg/kg)
Fe	0.55±0.01
Na	1.50±0.00
Ca	14.01±0.01
Mg	18.99±0.02

*Value reported as Mean ± SD for triplicate analysis n = 3*

**Table 5.** Result for Heavy Metal Concentration of *Fragaria x ananassa*

Elements	Concentration (mg/kg)	WHO/FAO permissible limit (mg/kg) (WHO 2016)
Cd	0.02±0.01	0.20
Pb	0.01±0.00	0.30
Cr	0.31±0.01	0.25
Pt	0.41±0.01	3.00

*Value reported as Mean ± SD for triplicate analysis n = 3*

**Table 6.** Result for Amino acid constituent of *Fragaria x ananassa*

Constituent	Concentration (mg/ml)
Tryptophan	0.0084±0.0001
Threonine	0.0196±0.0001
Isoleucine	0.0135±0.0002
Leucine	0.0326±0.0002
Lysine	0.0242±0.0003
Methionine	0.0025±0.0003
Glutamic acid	0.0952±0.0003
Phenylalanine	0.0184±0.0003
Tyrosine	0.0227±0.0002
Valine	0.0184±0.0003
Arginine	0.0255±0.0002
Histidine	0.0122±0.0001
Alanine	0.0319±0.0001
Aspartic acid	0.1493±0.0002
Cysteine	0.0065±0.0002
Glycine	0.0245±0.0002
Proline	0.0193±0.0002
Serine	0.0233±0.0002

*Value reported as Mean ± SD for triplicate analysis n = 3*

## CONCLUSION

The study confirms that strawberries from Plateau State are nutritionally rich, characterized by significant levels of carbohydrates, fibres, essential minerals, and bioactive compounds. Overall, the findings substantiate the nutritional and therapeutic value of *Fragaria × ananassa*, positioning it as a promising candidate for functional food development. However, the slight chromium contamination and the high ash content observed necessitate improved environmental monitoring and management to prevent potential contamination from soil or agricultural inputs. Ensuring food safety, while

promoting local cultivation, consumption, and processing of strawberries, could offer substantial health and economic benefits.

## ACKNOWLEDGEMENT

If not applicable, please expunge this section.

## CONFLICT OF INTEREST

Authors declare no conflict of interest.

## ETHICAL STATEMENT

This work required no ethical statement.

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