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Corrosion Inhibition and Adsorption Characteristics for Mild steel by Extract of Kyllinga Erecta Plant in Acidic Medium

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Abstract: The effectiveness of an ethanolic extract from Kyllinga erecta plant (KEP) as a Plant-derived corrosion inhibitor for mild steel in 0.5 M HCl was examined via the gravimetric analysis method across 298–318 K. KEPE markedly decreased the Surface degradation rate while enhancing the Inhibitory performance and concentration decrease in temperature. At 7 g/L, inhibition reached ~88 % at 303 K and 63 % at 318 K, demonstrating strong protective capability. Thermodynamic analysis, based on the Langmuir adsorption isotherm, provided insights into adsorption behavior. The calculated Gibbs free energy of adsorption (ΔG°_{ads}), activation energy (E_a), and heat of adsorption (Q_{ads}) confirmed a physisorption mechanism, implying electrostatic bonding between Kyllinga erecta plant extract and the metal interface. This protective barrier efficiently resists acid-induced metal deterioration. Phytochemical analysis identified bioactive compounds such as tanins, steroids, essential oil, flavonoids and chalcones, which contribute to corrosion inhibition. These compounds contain electron-donating functional groups that enhance adsorption and film formation. The eco-friendly, highly efficient, and readily available nature of Kyllinga erecta plant makes it a promising alternative to synthetic inhibitors, which often pose environmental and health risks.

Keywords: Kyllinga erecta, Mild Steel, Gravimetry measurement, Langmuir Adsorption Isotherm.

Introduction

Mild steel is a valuable and extensively utilized substance across multiple industries, particularly in construction, engineering, automotive, and chemical processing, attributable to its exceptional mechanical performance affordability, and ease of fabrication[1-2]. Its strength, weldability, and versatility make mild steel indispensable for manufacturing essential components such as pipelines, structural frameworks, boilers, heat exchangers, and machinery parts [3]. However, despite these advantages, mild steel's susceptibility to corrosion in harsh environments poses a significant challenge, leading to equipment degradation, safety hazards, and substantial economic losses for sectors employing steel-based facilities[5-6].

Corrosion is an inevitable and spontaneous electrochemical process that causes metals to deteriorate over time through reactions with environmental elements, including moisture, oxygen, and aggressive chemicals such as acids and bases [7]. In industrial settings, this process is accelerated by conditions like severe pH variations, thermal instability, and aggressive ions making corrosion prevention a critical issue. Mild steel corrosion results in the formation of iron oxides, leading to material weakening, structural failure, and increased maintenance and replacement costs [8]. Globally, the financial burden of steel corrosion is considerable, with annual costs running into billions of dollars due to maintenance, repairs, production downtime, and associated environmental and safety risks [9]. Thus, developing effective corrosion inhibition strategies for mild steel is essential, particularly accross fields such as naval, energy extraction, and industrial chemical production, where metal structures are exposed to aggressive environments [10].

Corrosion inhibitors offer a practical and cost-effective solution for mitigating metal degradation. Adhering to the metal, developing a defensive film that inhibits interaction with aggressive species, thereby slowing or halting the corrosion process[11-12]. Although numerous synthetic corrosion inhibitors have demonstrated effectiveness across various environments, many are toxic, expensive, and environmentally hazardous, leading to a growing demand for eco-friendly alternatives. This shift has driven interest in green inhibitors derived from natural resources, which offer biodegradability, low toxicity, cost-effectiveness, and availability making them particularly suitable for industries adopting sustainable practices [13-15].

Recent research has shown that plant extracts, rich in bioactive compounds containing sulfur, oxygen, nitrogen, and phosphorus, can function as potent anti-corrosion agents. These factors promote the binding to the metal surface, creating a thin defense layer that isolates the metal from destructive species [16]. Plant-based inhibitors have demonstrated high inhibition efficiency comparable to synthetic counterparts, making them a viable alternative [17]. The active components in these extracts, including alkaloids, flavonoids, terpenoids, and tannins, are believed to interact with the metal surface through electron donation and adsorption mechanisms, reducing corrosion [18].

Kyllinga erecta, a perennial sedge from the Cyperaceae family. This plant, commonly found in damp grassy areas worldwide, is known for its medicinal properties and traditional use. In Nigeria, it is locally referred to as "aayaaayaa" by the Hausa and "dogbodogbo" by the Yoruba [19]. The bioactive compounds present in *Kyllinga erecta* plant are hypothesized to offer corrosion inhibition by forming a protective membrane on the mild steel surface in acidic atmospheres.. This aligns with the global trend of investigating plant-based, eco-friendly corrosion inhibitors, which are increasingly recognized for their efficacy and environmental compatibility [20]. Therefore, in this study *Kyllinga erecta* plant was taken to investigate the inhibitive effect for corrosion of mild steel in 0.5M HCl.

Materials and Method

Collection and Preparation of Plant Extract

Kyllinga erecta plant was gathered from the botanical garden of Federal polytechnic, Ilaro. Following collection, the plant was carefully washed with distilled water and permitted to air dry in a well-ventilated area for about three weeks. Once dried, the plant material was ground into a fine powder. A specific quantity of the powdered sample was soaked in ethanol for 48 hours, after which the mixture underwent filtration with Whatman filter paper, and the filtrate was then subjected to concentration using a rotary evaporator to remove the solvent, producing a concentrated stock solution.

Preparation of Metal Coupons

The metal coupons of 1.2 × 4.0 × 0.3 cm were used for mass loss measures while 1 x 1cm specimens were used for electrochemical analysis. After mechanical polishing, the samples were cleaned with double distilled water, degreased by acetone, and then the samples were used in weight loss tests and surface analysis.

Gravimetry Corrosion Experiment

In gravimetric corrosion experiment, mild steel was drilled and cut into individual coupons, each with dimensions of 20 x 20 x 1.1 mm. These coupons were degreased by washing with ethanol, rinsing with distilled water, and finally cleaning with acetone to ensure quick drying before storage in a desiccator. Each coupon was initially weighed and then immersed in 30 ml of test solution with varying concentrations (1.0, 2.0, 3.0, 4.0, 5.0, 6.0 and 7.0 gL^{-1}) were prepared with 0.5M HCl, in 100 ml beakers. The beakers were then placed in a water bath set at a constant temperature of 298K, and the immersion time ranged from 1 to 7 hours. At each hour, the metal coupon was removed, washed with distilled water and rinsed with acetone and reweighed [21-22]. This process was repeated at 308K and 318K. the weight loss was calculated, the inhibition efficiency, and corrosion rate of mild steel were determined using the equation below.

CR = $\frac{\Delta W}{At}$ …………………………………..(1)

%IE = (1- $\frac{W1}{W2}$) × 100……………………....(2)

Results and Discussion

Impact of Inhibitors Concentration

The results on the protective effectiveness of mild steel in 0.5M HCl at room temperature using varying concentrations of *Kyllinga erecta* plant extract are presented in Table 1. It is evident from the experimental outcome that the corrosion rate is greatly reduced with the increase in concentration of the inhibitor, meaning that the corrosion resistance has certainly enhanced. This implies that the inhibitor molecules are likely to attach to the interface of the mild steel, creating a shield that decreases the available active sites, which are susceptible to corrosion, greatly diminishing their numbers. Therefore, the mild steel becomes increasingly acceptable to a hostile environment without corrosion [20].

After 7 hours of immersion, the most efficient protection of 88% was obtained with 7 grams of KEPE per liter. Because of the high level of protection, KEPE binding affinity with mild steel surface is strongly evident. As the high level of protection suggests, this indicates the ability of KEPE to function as a benign inhibitor of deterioration of mild steel in strongly acidic media [22]. Additionally, the results indicate that perhaps other factors such as the amount of time the surface is submerged or the steel's surface characteristics can also alter the effectiveness of the inhibitor. [23].

Gravimetric Measurements

The comment method which reviews inhibition of corrosion by measurement of weight loss is accepted as one of the most accurate methods because it is easy to perform. Thus, it is a basic method for many programs concerned with corrosion monitoring. This particular investigation involved weight loss assessments carried out at temperatures of 298K, 308K, and 313K with a KEPE in a chloride acid solution conducted over a broth immersion of 7 hours. This study was staged to define the effect of varying KEPE in concentration of mild steel 0.5 M hydrochloric acid solution.

The figures together with the table clearly depict the decrease in the corrosion rate together with inhibition efficacy as higher amount of plant extract of *Kyllinga erecta* is used. This is suggestive of interplay between the extracts phytochemical constituents and the mild steel interface which gives rise to an ameliorated protective layer. 88.14% The greatest inhibition performance at 7g/L concentration together with temperature of 303K. This phenomenon is attributed to a particular adhesion process, whereby bioactive constituents from the extracts attach to the interface of the metal[23-24] and impede the functional sites that catalyze the corrosion process.

Effect of Temperature

The gravimetric evaluations were carried out at temperatures spanning from 298 K to 318 K.. Thermodynamic variables Were leveraged to explore the role of temperature, important for understanding the resilience of the inhibitor layer on the metal interface. The findings indicated that the efficacy of inhibition diminishes (i.e. from 88.14-67.25%) as the temperature rises (i.e. from 318-298K). In acidic environments, the generation of hydrogen gas typically enhances the electrochemical deterioration, leading to an increased corrosion rate [21].

Table 1: Corrosion characteristics of Mild steel exposed to a 0.5 M HCl solution with and without changing concentrations of KEPE-based on weight loss analysis over 7 hours at varying temperature.

Immersion period	Concentration of inhibitor (gL^{-1})	298K		308K	
		CR (mmpy)	IE (%)	CR (mmpy)	
7 hr	BLANK	58.34	-	426.34	-
	1	19.32	66.88	209.56	50.85
	2	17.91	69.30	167.45	60.72
	3	11.23	80.75	121.89	71.41
	4	10.12	82.27	112.26	73.67
	5	8.46	85.50	101.21	76.26

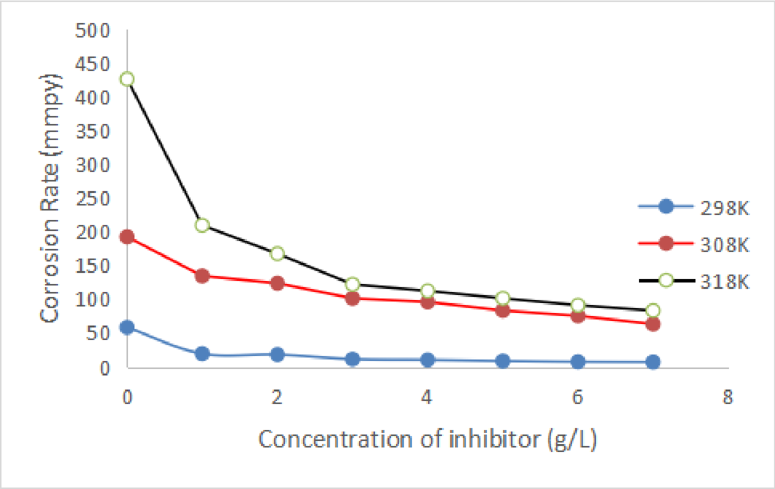


Figure 1. Amount of inhibitor (g/L) and corrosion rate (mmpy) of mild steel in varying concentrations of KEPE at varying temperatures in a 0.5M HCl solution.

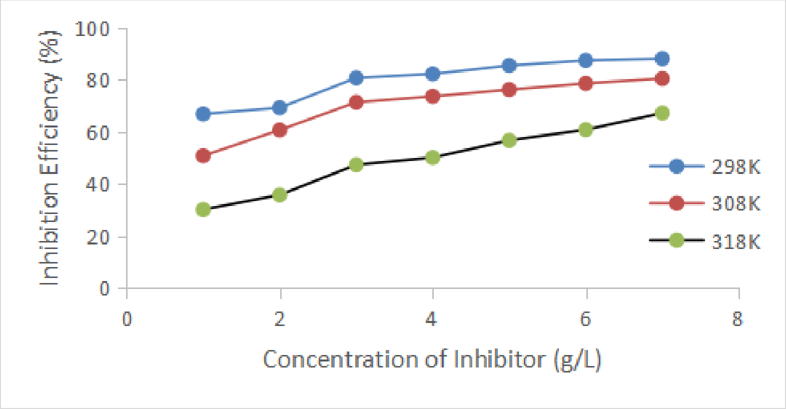


Figure 2. The inhibitor dosage (g/L) and corrosion inhibition performance (%IE) of mild steel in varying concentrations of KEPE at different temperatures in a 0.5M HCl solution.

Adsorption Isotherm

Langmuir isotherm model best describes the phenomenon when alloyed steel surfaces are covered in protective coating of the inhibitor compounds; it explains how the affinity of the metal for the inhibitor's adsorbing atoms is strong enough to allow them to bond to the surface, thus effectively preventing the corrosion process. Adsorption isotherms provide one of the simplest mathematical representations of the adsorption process. These equations relate the amount of adsorbate on a surface to the concentration of the adsorbate in the surroundings[25]. The following equation can be used to represent Langmuir isotherm.

$$\frac{C}{\theta} = \frac{1}{\mathrm{Kads}} + C \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3)$$

C represent the inhibitor's concentration, θ for the percentage of the surface covered, and K_{ads} for the adsorption coefficient. The analysis and straight-line fitting of the connection between C/θ and C in Figure 5 produced regression coefficients of 0.9974 at 298 K, 0.9991 at 308 K and 0.9698 at 318 K. Strong evidence that the adsorption process follows the Langmuir adsorption isotherm is provided by these values, which are almost identical to one. The following formula was used to calculate the free energy of adsorption, or G_{ads}, from the equilibrium constant of adsorption:

$\Delta G^{o}_{ads} = -RTln(55.5K_{ads})$ ………………………… (4)

T denotes the absolute temperature, R stands for the universal gas constant, and 55.5 represents the molar concentration of water in the solution. It is widely recognized that physisorption, driven by Coulombic interactions between charged inhibitor molecules and the charged metal surface, is characterized by ΔG_{ads} values of 20 kJ/mol or lower. Conversely, ΔG_{ads} values of -40 kJ/mol or higher signify chemisorption, which involves the transfer or sharing of charges between organic molecules and the metal surface, leading to the formation of a coordinate-type metallic bond. From the Langmuir plot's intercept, the average K_{ads} value was determined to be 1.08 g/L (refer to Table 2). The ΔG_{ads} values obtained in this study were 5.20 kJ/mol at 318 K, 11.23 kJ/mol at 308 K, and 16.32 kJ/mol at 298 K. These results suggest that the adsorption process is predominantly physisorption, indicating that the formation of an inhibitor molecule layer on the metal surface effectively mitigates corrosion [26]. The negative ΔG_{ads} values further confirm the spontaneous adsorption of inhibitor molecules onto the metal surface. The ΔG_{ads} data at all temperature are shown in table 2. The (ΔH_{ads}) was measure agreeing to the Van't Hoff equation

$Log\ K_{ads} = -\Delta H_{ads}/2.303RT + constant$ ……………………………….. (5)

The ΔS_{ads} data was calculated and recorded in table 3. Then by applying the next balance:

$\Delta G_{a}d_{s} = \Delta H_{ads} - T\Delta S_{ads}$ ………………………………………………….. (6)

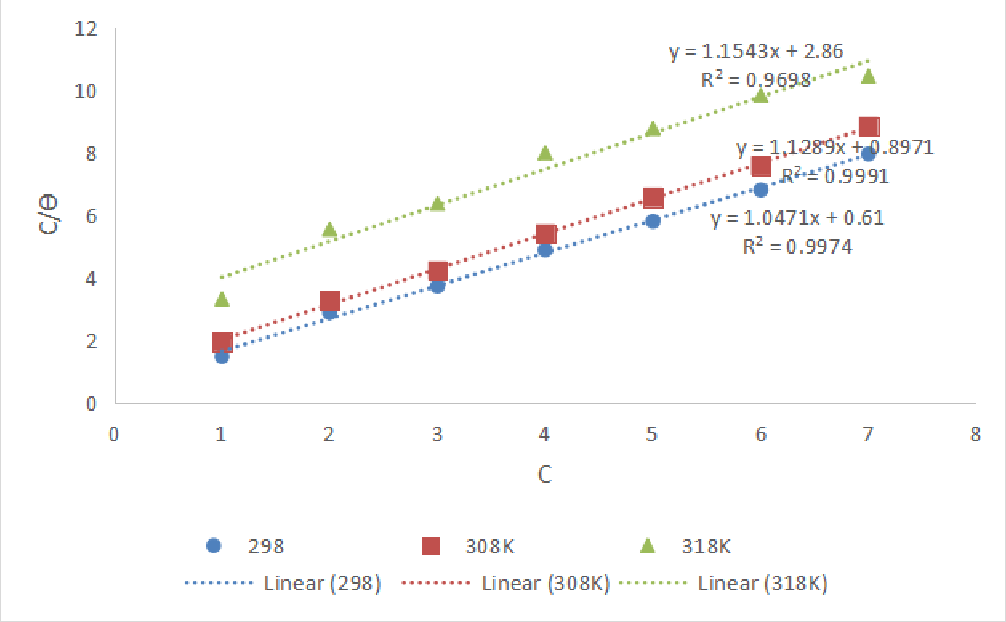
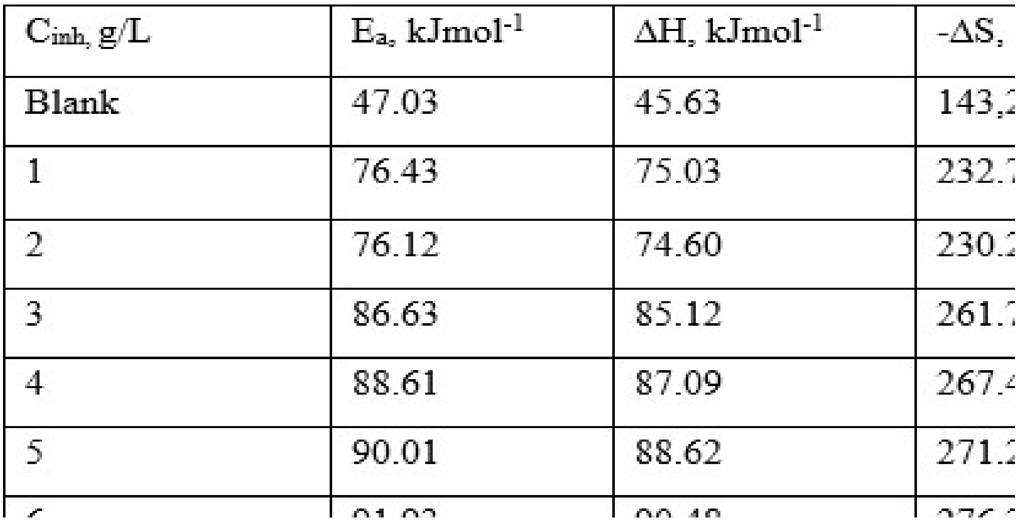


Figure 3. Langmuir adsorption isotherm plot for the adsorption of different concentration of KEPE on the surface of Mild Steel in 0.5 M HCl solution for 7 hours at varying temperatures.

Table 2: Langmuir adsorption constant and change in free energy at different temperature.

Table 3. Activation parameters (Ea, ΔH and ΔS) for Mild Steel in the presence and absence of different concentration of KEPE.



C_{inh}, g/L	E_a, kJmol^{-1}	ΔH, kJmol^{-1}	-ΔS,
Blank	47.03	45.63	143,
1	76.43	75.03	232.
2	76.12	74.60	230.
3	86.63	85.12	261.
4	88.61	87.09	267.
5	90.01	88.62	271.
[illegible]	[illegible]	[illegible]	[illegible]

FTIR Analysis of *Kyllinga erecta* Plant Extract

The analysis of corrosion-inhibiting components in the extract was conducted using FTIR (Fourier transform infrared spectroscopy), a method employed to detect functional groups within molecules. Figure 5 illustrates the FTIR spectra of KEPE and its adsorption on a mild steel substrate. In the FTIR spectrum of the KEPE, the peak observed at 452 cm^{-1} is attributed to the bending vibration of the C-H bond. A peak at 600 cm^{-1} is associated with either C-C-C bending vibrations or C-O out-of-plane bending vibrations in aromatic rings. Additionally, the signal at 834 cm^{-1} corresponds to C-H or C-O-C bending vibrations. Peaks below 1000 cm^{-1} are linked to aliphatic or aromatic C-H bonds. These findings suggest that the KEPE contains a variety of functional groups, indicating its potential as an effective corrosion inhibitor [23-26].

Further analysis reveals that the peak at 1046 cm^{-1} is indicative of C-O stretching vibrations (primary or secondary alcohol) or C-N stretching vibrations (amine). The peak at 1422 cm^{-1} is attributed to C-H bending vibrations in alkane groups or C-O-H bending vibrations in carboxylic acids. A prominent peak at 3298 cm^{-1} corresponds to O-H or N-H stretching vibrations, characteristic of hydroxyl and amine groups, respectively. The peak at 2322 cm^{-1} is associated with O-H stretching vibrations (hydrogen-bonded) or C≡C stretching vibrations in alkynes. Lastly, the signal at 2910 cm^{-1} is related to C-H stretching vibrations in alkanes. These observations collectively highlight the presence of diverse functional groups in the extract, supporting its potential as a corrosion inhibitor.

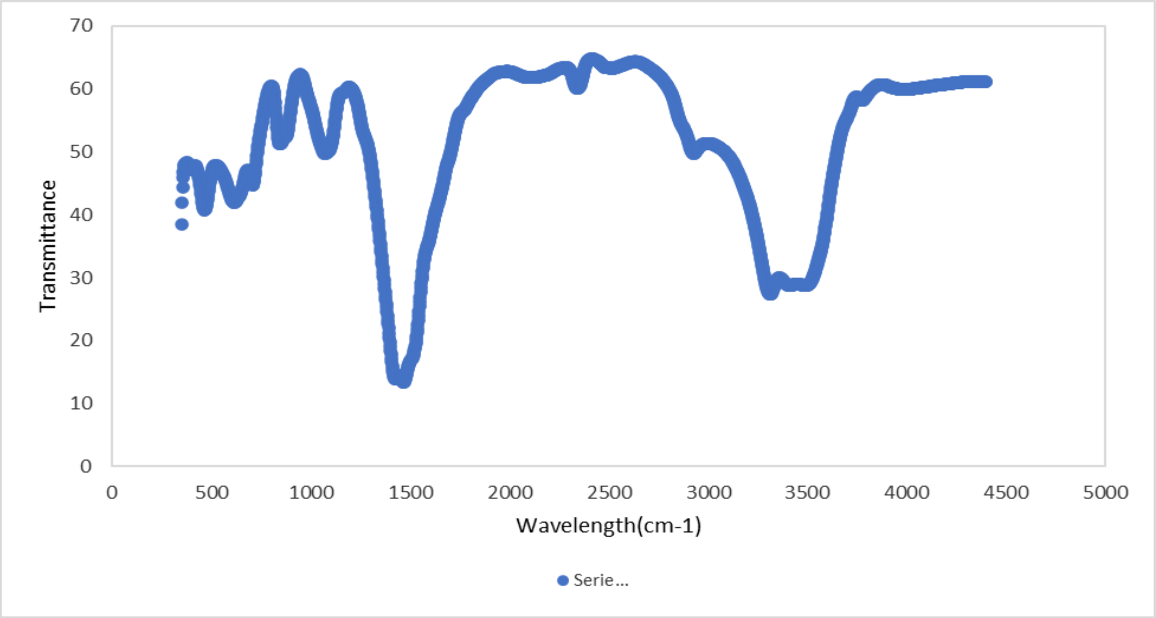


Figure 4. FTIR Analysis of KEPE

Surface Morphology

The mild steel surface, after being immersed in 0.5 M HCl for 5 hours at 25 °C, was examined using scanning electron microscopy (SEM) to assess the impact of KEPE at a concentration of 1 g/L. As shown in Fig. 5, the steel interface directly exposed to the acid displayed minimal roughness, indicating significant corrosion activity caused by the acid. The corrosion damage was uniform, with no clear signs of localized corrosion. In contrast, the addition of KEPE led to a noticeable reduction in surface roughness, suggesting that the extract effectively reduced the corrosion process. This protective effect is likely due to the development of a barrier layer on the metal interface, which limits the penetration of corrosive agents. Additionally, slight traces of the extract were observed on the steel surface, further supporting its role in corrosion mitigation[16-18].

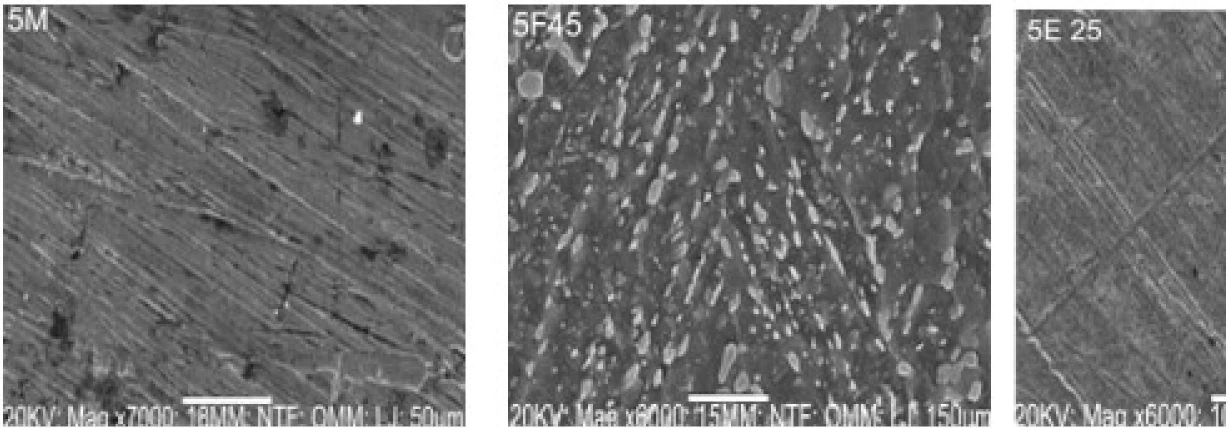


Figure 5. Scanning Electronic Microscopy (SEM)-images of Mild Steel (MS) (X) Ordinary MS (Y) MS in 0.5M HCl Solution (Z) MS in the presence of KEPE.

EDX Results

The Energy Dispersive X-ray (EDX) technique was utilized to analyze the surface composition of mild steel submerged in a 0.5 M HCl solution, with and without an KEPE. The EDX spectra related to these two situations are shown in Figure 8. Without the inhibitor, the iron (Fe) content measured at 74.60%, but it dropped to 63.50% upon the application of the optimal concentration of KEPE. The spectra shown in Figure 8 show a notable decrease in the Fe peaks for the sample that was treated with the inhibitor in comparison to the untreated sample. This reduction indicates the creation of a protective film on the mild steel interface due to the inhibitory effect[23].

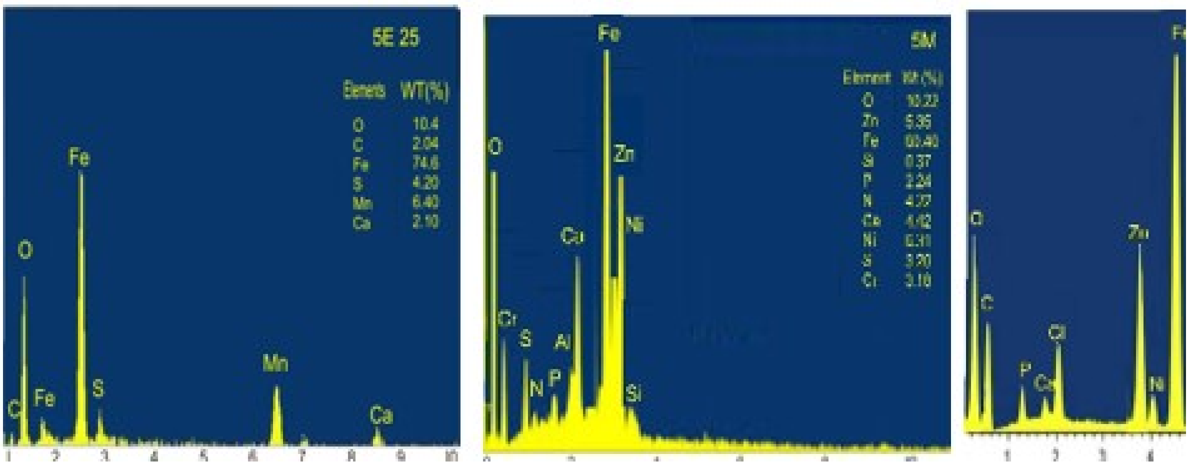


Figure 6. X-ray Diffraction (XRD)-images of Mild Steel (MS) (A) Ordinary MS (B) MS in 0.5M HCl Solution (C) MS in the presence of KEPE.

Conclusion

The KE plant extract has shown to successfully prevent the corrosion of mild steel in a 0.5 M hydrochloric acid (HCl) solution. Gravimetric analyses showed that the inhibition effectiveness reached 88% with a minimal extract concentration of 7 g/L. It was noted that effectiveness enhanced with greater extract concentrations but declined as the temperature increased. The inhibitor species' adsorption features were determined to correspond with the Langmuir isotherm model, and the computed free energy of adsorption suggested a spontaneous process, verifying the bonding of the inhibitor species to the mild steel surface. A chemisorption mechanism was suggested for the interaction of KEPE species with the steel surface in a 0.5 M HCl solution,corroborated by the connection between extract adsorption and solution temperature. This mechanism was additionally supported by the determined activation energy (Ea) and heat of adsorption (Q_{ads}) values, which offered further evidence for the corrosion inhibition behavior noted [26-27].

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