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Contribution of Environmentally friendly inhibitor in protection of aluminum alloy in acidic Solution

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ABSTRACT

The utilization of plant extracts is an efficient alternative to conventional corrosion inhibitors since they play a vital role in minimizing corrosion risks and are a natural and eco-friendly source of inhibitors. The aim of this research is to investigate the potency of jujube leaves as a natural green inhibitor for corrosion of aluminium alloy immersed in nitric acid (1M). The extracts were tested at different temperatures (40, 50, 60 and 70°C) and different inhibitor concentrations (0, 30, 60, 90 and 120 ml/L). According to the study, in weight loss analysis, a maximum inhibition efficiency of 92.43 was observed at 40 oC at an inhibitor concentration of 120 ml/L. The findings validate the capability of Zizphus leaves extract to reduce corrosion rates. The efficient performance was due to the presence of active organic compounds in the leaves, which form a protective layer not allowing the direct interaction of metal with acid.

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1. INTRODUCTION

Corrosion is a serious challenge faced by industries with a direct and serious impact upon the structural integrity of the infrastructure and the operational processes [1,2] and moreover causing economic losses of billions of dollars annually. So, corrosion can be defined scientifically as a natural process wherein metals try to achieve the most stable and consistent form of nature. Naturally unstable metals will react with the environment and form oxides, hydroxides and sulfides. This mineral decomposition occurs when chemical and/or electrochemical reactions take place with elements in the surrounding environment [3]. Corrosion has extremely serious negative repercussions, leading to the deterioration of the mechanical and structural properties of materials, including weakening their strength, distorting their appearance, and increasing their permeability to liquids and gases. Therefore, corrosion is classified as a major crisis in various industrial fields, such as power plants, chemical reactors, household appliance manufacturing, and material transport pipelines, as well as the arms and bridge sectors [4]. The engineering challenge is particularly pronounced when dealing with aluminum alloys in acidic environments. This alloy is widely used in aerospace and construction applications [5]. As a result, awareness has recently grown regarding the need to develop effective methods for preventing corrosion to reduce the material and human damage it causes [6,7]. To combat corrosion attacks and mitigate their impact, various technologies have been developed, such as coatings and cathodic protection. Corrosion inhibitors stand out as one of the most powerful and cost-effective technologies [8] in terms of results. Inhibitors are chemical substances added in very small quantities but achieve high efficiency in reducing the rate of metal corrosion. These substances are classified based on their chemical composition, physical properties, and mechanism of action [9,10].

Organic inhibitors are widely used due to their superior protective performance. Their principle is based on the adsorption of chemicals (physically or chemically) onto the metal surface, leading to the formation of a barrier layer that prevents direct contact between the metal surface and the corrosive solution. Despite the effectiveness of synthetic organic inhibitors, their chemical production processes are very expensive, and they are often toxic substances with harmful environmental effects [11,12]. Hence, significant research interest has emerged in finding natural "green" sources as environmentally friendly and inexpensive alternatives. Plant extracts are a vital resource for corrosion inhibitors due to their economic benefits and complete environmental safety. In this regard, it is established that jujube leaves are one of the most important plant sources that can be relied upon, given their unique biological and chemical properties, to serve as an effective and sustainable inhibitor [13]. Due to the presence of active compounds and the pronouncement of flavonoids, tannins and saponins (high concentration), Zizphus leaves possess these biological and chemical properties [14]. Zizphus leaves are known to be a good antioxidant [15, 16], with a greater power to scavenge free radical as a result of their chemical composition. Medicinal and therapeutic uses are also evident in jujube leaves; powdered jujube leaves are used as a natural wash to treat dandruff and strengthen hair follicles. From a scientific and research perspective, Zizphus leaves are classified as an ideal source for producing green corrosion inhibitors because they are non-toxic. Furthermore, Zizphus leaves form a protective layer due to their organic molecules having a high absorption capacity on the metal surface, thus forming a protective shield that prevents chemical corrosion.

2. Practical Part

2.1 Preparation of the Inhibitor (Ziziphus Leaves)

1. **Sample Collection**

Ziziphus leaves were collected from two local sources in Diyala Governorate: the first from a home garden and the second from the campus of the College of Engineering, University of Diyala. Healthy leaves were selected to ensure the quality of the extraction, as shown in Figure. 1, (a).

2. **Cleaning and Washing**

The samples were washed several times using only distilled water to ensure the removal of suspended particles and dirt without affecting the chemical composition of the leaves. Tap water was avoided to prevent any ionic contamination or impurities that could affect the accuracy of the results later, as shown in Figure.1, (b).

3. **Drying**

The washed leaves were placed in baskets with ventilation holes on all sides to ensure airflow, as shown in Figure.1, (c). The drying process was carried out in a dark, well-ventilated place at room temperature for a period of (12) days until complete drying. This method (shade drying) was chosen instead of direct sun drying to preserve the active and heat- and light-sensitive materials from decomposition.

4. **Grinding and Preparation**

After verifying that the leaves were completely dry, the samples were ground using a hand mill to obtain a fine powder. This was followed by sieving the powder to obtain a fine powder ready for laboratory preparation, as shown in Figure.1, (d), to ensure granular homogeneity.

Figure 1. Preparation steps of Ziziphus leaves used for inhibitor extraction: (a) fresh leaves after collection, (b)

washing with distilled water, (c) shade drying at room temperature, and (d) grinding and sieving to obtain fine powder.

2.2 Sample Preparation

Specimens of **AA2024-T3 aluminum alloy** were obtained from Baghdad, Bab Al-Muadham, Iraq. The chemical composition of the alloy was verified using **optical emission spectroscopy (OES)** to confirm its conformity with the standard specifications, and the obtained results showed full compliance.

The alloy was cut in the engineering workshop of the College of Engineering, University of Diyala, into rectangular specimens with dimensions of **5 cm × 1 cm × 3 mm**. A small hole was made near one end of each specimen to facilitate suspension during immersion in the corrosive medium, as shown in **Figure 2(a)** .

The specimen surfaces were then polished using fine sandpaper in a uniform circular motion until a smooth, scratch-free finish was obtained, as shown in **Figure 2(b)** . After polishing, the specimens were thoroughly cleaned with **methanol, acetone, and distilled water** in order to remove any surface impurities. Finally, the cleaned specimens were dried using a stream of hot air to ensure complete removal of moisture before use in the corrosion experiments, as shown in **Figure 2(c)** .

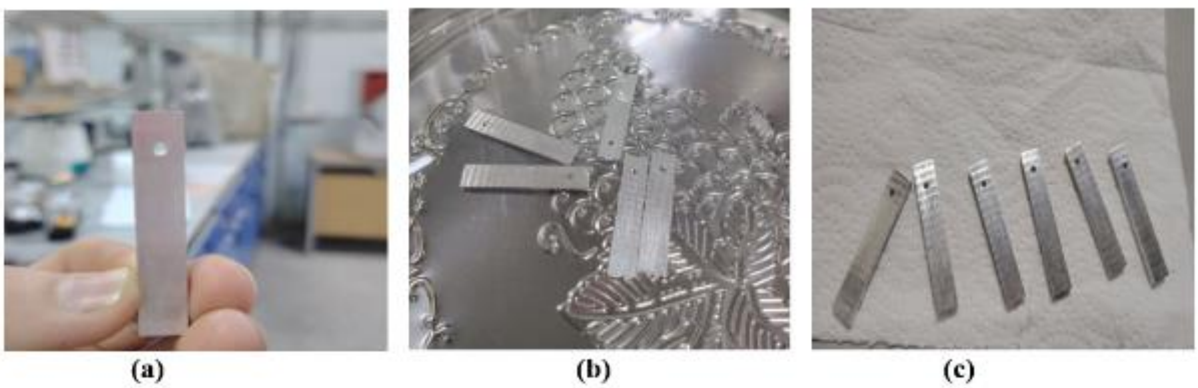
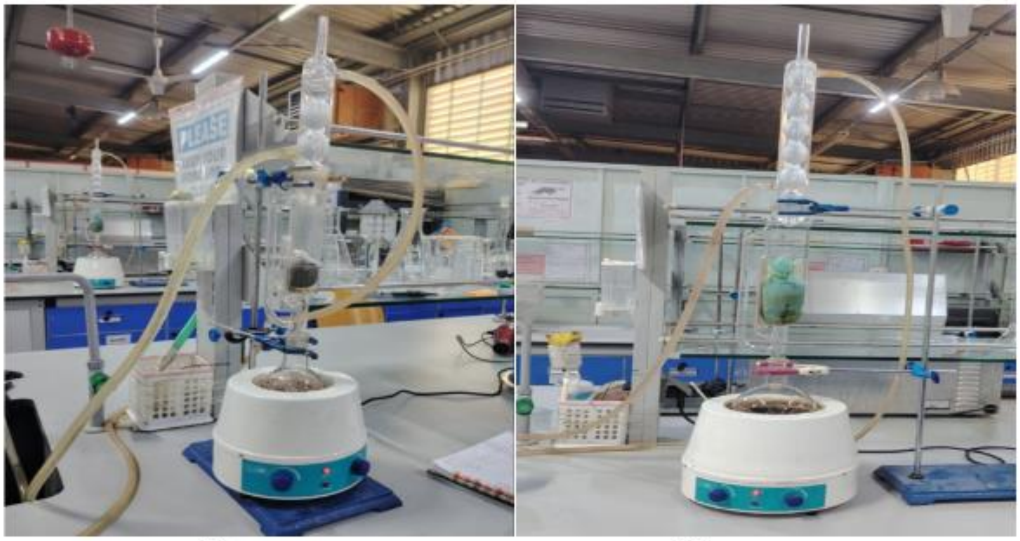


Figure 2. Preparation of AA2024-T3 alloy specimens for corrosion tests: (a) drilled specimen after cutting to the required dimensions, (b) polishing and surface finishing of the specimens, and (c) cleaned specimens ready for immersion experiments.

2.3 Soxhlet Extraction

The Soxhlet extraction technique was employed to obtain the active organic constituents from **Ziziphus leaves powder**, which were later used as a green corrosion inhibitor. Two different quantities of the powdered leaves were prepared according to the size of the extraction setup. In the larger Soxhlet apparatus, **50 g** of powdered leaves were used and packed in a porous medical mask, whereas in the smaller apparatus, **10 g** of powdered leaves were packed in small porous tea bags. The packed plant material was placed inside the extraction chamber of the Soxhlet apparatus, while **distilled water** was used as the extraction solvent in the round-bottom flask. Upon heating, the solvent evaporated, condensed in the condenser, and percolated through the powdered leaves in the extraction chamber. When the solvent level touched the top of the siphon arm it automatically fell back in the flask carrying along with it the solubles extracted. This cycle was repeated continuously until an adequate extraction was achieved, as in Figure 3. The extraction was done with distilled water for a period of time, 150 to 250 min depending upon the quantity of the herb and the size of the extractor.



(a) (b)

Figure 3. The Soxhlet extraction of the leaf powder of Ziziphus using distilled water was setup with 50 g powder in (a) the bigger apparatus, and 10 g powder in (b) the smaller apparatus.

2.3 Mathematical Relations (weight loss)

The corrosion behavior of AA2024-T3 aluminum alloy in nitric acid solution in the absence and presence of Ziziphus leaves extract as a corrosion inhibitor was evaluated using weight loss method. This method is extensively used for calculating corrosion rate, inhibition efficiency and surface coverage of metal samples. Prior to immersion, an accurate measurement took place of the dimensions of the aluminum alloy samples, and the initial weight of each one was measured with an analytical balance. Afterward, the samples underwent cleaning with acetone and distilled water. Subsequently, they were dried using an electric dryer before immersion. Following cleaning, the specimens were placed in the test solutions for 3 h under various experimental conditions, as depicted in Figure 4. The experiments were carried out at different inhibitor concentrations of 0, 30, 60, 90, and 120 mL/L, and at different temperatures of 40, 50, 60, and 70°C. At the end of each experiment, the specimens were removed from th The experiments were conducted with varying the concentration of the inhibitor at 0, 30, 60, 90, and 120 mL/L. The temperature at which the experiment was conducted was 40, 50, 60, and 70 °C. Once the experiments were completed, the specimens were taken out of the test solutions and cleaned well by washing with ethanol, acetone and distilled water, and dried again in an elettric dryer. The same analytical balance was used to weigh each specimen at the end. The test solutions were washed in ethanol, then in acetone, then in water distilled and dried a second time in an electric drier. The measurements of each specimen's final weight were taken on the same analytical balance.

The difference in weight at the start and the end was taken for the calculation of weight loss of alloy specimens. By means of the values attained, the corrosion rate (CR), Inhibition efficiency (IE%) and surface coverage (θ) were evaluated from the equations shown in the part of Mathematical Relations.

$$CR = \frac{W}{A \times t}$$

$$IE\% = \left(1 - \frac{CR}{CR^0}\right) \times 100$$

$$\theta = \frac{IE\%}{100}$$

Where:

- **CR** is the corrosion rate in the presence of inhibitor, expressed in **g m^{-2} day^{-1}**.
- **CR^0** is the corrosion rate in the absence of inhibitor.
- **W** is the weight loss of the aluminum alloy specimen before and after immersion, expressed in **g**.
- **A** is the total surface area of the specimen, expressed in **m^2**.

- **t** is the immersion time, expressed in **days**.
- **IE%** is the inhibition efficiency.
- **θ** is the surface coverage.

Figure 4. The water bath used for measuring the weight loss of AA2024-T3 aluminum alloy specimens immersed in nitric acid solutions with and without an inhibitor.

3. Results and Discussion

3.1 Yield of extraction process

Table 1. Extraction conditions and percentage yield of Ziziphus leaves extract.

Run	Time(min)	Powder mass(g)	Solvent Volume(ml)	No. of Cycle	Yield (%)
1	200	15	150	21	10.3
2	200	50	500	11	11.1
3	190	10	100	14	10.5
4	210	40	400	9	8.3
5	200	15	150	13	10
6	218	40	400	8	7.2
7	150	15	150	15	9.5
8	210	50	500	14	7.5
9	203	15	150	16	10.2
10	250	40	400	14	7.6
11	180	15	150	12	9.0
12	230	40	400	14	11.8
13	160	10	100	16	10.5

Table 1 presents the extraction conditions along with percentage yield from Soxhlet extraction of Ziziphus leaves powder. The calculation of extraction yield was done using following formula.

$$Y\% = \left(\frac{m_{p(f)}}{m_p}\right) \times 100$$

The percentage yield of extraction Y% is defined as m_{p(f)}/m_p m_{p(f)} is the mass of extract obtained after extraction and m_p is the mass of powdered leaves (used for extraction) before extraction.

The results showed an extraction yield between 7.2% and 11.8%, depending on the extraction conditions. The extraction conditions include extraction time, powder mass, solvent volume, and number of cycles.

3.2 Corrosion Rate and Inhibition Efficiency of Ziziphus Leaves Extract in 1 M HNO_3

The weight loss technique is employed for the comparative study of AA2024-T3 aluminium alloy corrosion in 1M HNO3 without and with inclusion of different concentrations of Ziziphus leaves extract as a green corrosion inhibitor. As seen in Table 2, the corrosion rate and inhibition efficiency are dependent upon inhibitor concentration and temperature.

Table 2. The impact of inhibitor concentrations and temperature on the corrosion rate and inhibition efficiency of a 2024-T3 aluminum alloy in 1 M HNO_3.

Inhibitor Concentration (ml/L)	Temperature (C)	Corrosion rate (gmd)	%IEWL
0	40	191.78	---------
30		55.405	71.11
60		43.831	77.15
90		28.843	84.96
120		13.750	92.83
0	50	493.15	--------
30		154.997	68.57
60		120.820	75.5
90		82.849	83.20
120		56.367	88.57
0	60	1041.09	---------
30		361.674	65.26
60		289.318	72.21
90		209.571	79.87
120		163.867	84.26
0	70	1467.12	-------
30		630.568	57.02
60		469.478	68.00
90		380.570	74.06
120		338.904	76.90

As seen in Table 2, the corrosion rate of AA2024-T3 aluminum alloy increased significantly with temperature in the uninhibited 1 M HNO_3 solution, suggesting that higher temperatures accelerate the corrosion process. On the other hand, Ziziphus leaves extract reduced the corrosion rate significantly at all studied temperatures, confirming its inhibitive effect.

It is apparent that with increasing inhibitor concentration from 30 to 120 mL/L in every temperature, inhibition efficiency increased. The inhibition efficiency was the highest at 120 mL/L and 40°C (92.83%) and gradually decreased as the temperature increased up to 70°C (76.90% at 120 mL/L and 70°C). With increased temperature, it appears that the protective action of the inhibitor is weakened due to being less adsorbed on the alloy surface.

3.2.1 Effect of Temperature on the Corrosion Rate of AA2024-T3 Aluminum Alloy in 1 M HNO_3 for 3 h

The impact of temperature on the corrosion rate of an AA2024-T3 aluminum alloy in 1 M HNO_3 for consecutive immersion times of 3 h is demonstrated in Figure 5. The corrosion rate increased markedly with temperature, from 191.78 g m^{-2} day^{-1} at 40°C to 1467.12 g m^{-2} day^{-1} at 70°C.

The elevation in temperature may accelerate electrochemical reactions and the dissolution of alloy surface, eventually increasing the ionic mobility of the corrosive medium and thus, enhancing the corrosion of the alloy. Moreover, the increase in temperature may compromise the stability of the naturally formed oxide film on the aluminum alloy surface, limiting its protective effect and causing the alloy to corrode.

Overall, the results indicate that increasing the temperature has a pronounced effect on accelerating the corrosion process of AA2024-T3 aluminum alloy in 1 M HNO_3.

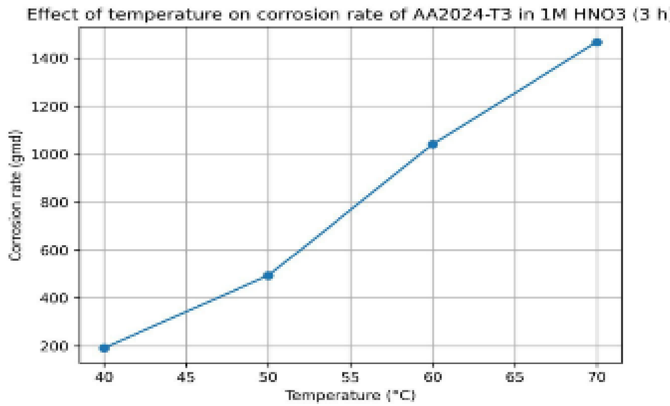


Figure 5. Effect of temperature on the corrosion rate of AA2024-T3 aluminum alloy in 1 M HNO_3 for 3 h.

As evident from Figure 5, the temperature increment increases the rate of corrosion. Owing to the improvement of electrochemical reactions and a decrease in the stability of the oxide layer on the alloy surface, higher temperature has been realized. This indicates that the AA2024-T3 aluminum alloy undergo corrosion at a faster rate as temperature increases in 1 M HNO_3.

3.2.2 Impact of Inhibitor Concentration on the Corrosion Rate of Aluminum Alloy AA2024-T3 in 1 M HNO_3 for 3 Hours

The graph in figure 6 shows the influence of Ziziphus leaves extract concentration on the corrosion rate AA2024-T3 aluminum alloy in 1 M of HNO_3 at the temperature: 40°C, 50°C, 60°C, 70°C. Clearly, the corrosion rate decreases with increasing inhibitor concentration at all the temperatures studied.

The adsorption of active Ziziphus leaves extract constituents may have occurred on the alloy surface, namely phenolic and flavonoid compounds. The adsorbed molecules form a protective film which decreases the exposure of the metal surface to the acidic medium and hence reduces the rate of corrosion. Generally, the rising of the concentration of the inhibitor was found to increase the performance of the extract.

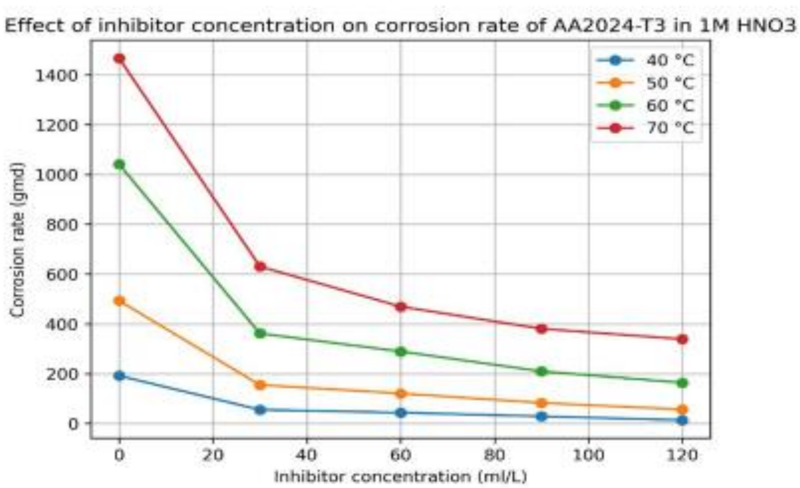


Figure 6. The concentration of inhibitors impacts the corrosion rate of AA2024-T3 aluminium alloys in 1 M HNO_3 in 3 h at different temperatures.

As shown by Figure 6, corrosion rate was seen to decrease with increasing inhibitor concentration at all temperatures (40, 50, 60 and 70°C). The adsorption of the active components of Ziziphus leaves extract (phenolic and flavonoid compounds) on the ZM9 alloy surface explains its behavior. When the adsorption takes place, a film is formed which limits the contact of the metal surface and the corrosive acidic solution. This results in reduced corrosion rate.

3.2.3 Effect of Ziziphus Leaves Extract Concentration on the Inhibition Efficiency of AA2024-T3 Aluminum Alloy in 1 M HNO_3

The inhibition performance of Fini 92K Ziziphus leaves extract on AA2024-T3 aluminum alloy in 1M HNO_3 at different temperatures is shown in figure 7. The results show that increasing the inhibitor concentration increases the inhibition efficiency at all investigated temperatures. This behavior may be due to the increased adhesion of the active constituents of the extract onto the alloy surface at higher inhibitor concentration. Therefore, the compactness of adsorption of the inhibitor inhibits the entry of the corrosive medium into the metal surface protecting it efficiently owing to which the inhibition performance improves. The efficiency of inhibition was maximum at maximum inhibitor concentration while this efficiency generally decreased with an increase in temperature.

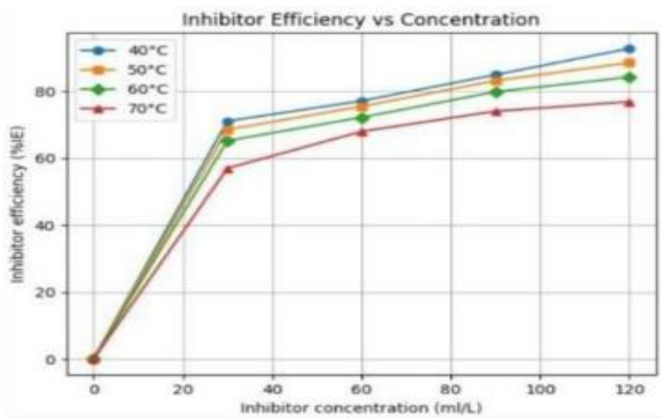


Figure 7. The Impact of Ziziphus leaves extract concentration on the Inhibition efficiency of AA2024-T3 aluminium alloy in 1 M HNO_3 at different temperatures.

3.2.4 Effect of Temperature on the Inhibition Efficiency of Ziziphus Leaves Extract for AA2024-T3 Aluminum Alloy in 1 M HNO_3

As illustrated in Figure 8, the rise in temperature enhances the inhibitory efficiency of Ziziphus leaves extract for aluminum alloy AA2024-T3 in 1 M HNO_3 solution at various inhibitor concentrations. All the concentrations studied were found to show a decrease in inhibition efficiency with rise in temperature.

The alteration may be because adsorbed inhibitor layer stability decreases with temperature rise. With the rise of temperature, some of the inhibitor molecules started partially getting desorbed from the alloy surface. This reduces the protective effect of the adsorbed film, thus permitting greater interaction between the metal surface and corrosive medium. As a result, at higher temperatures the inhibition efficiency of the extract becomes less effective.

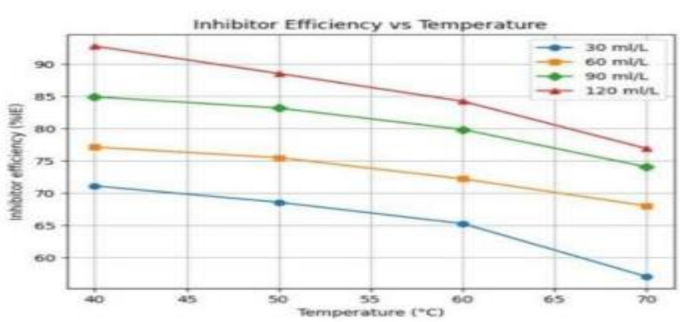


Figure 8. Impact of the temperature on the inhibition efficiency of the extract of Ziziphus leaves on AA2024-T3 aluminum alloy in 1 M HNO_3 at various inhibitor concentrations

3.3 Optical Emission Spectroscopy (OES) Analysis

Optical emission spectroscopy (OES) was used to determine the chemical composition of the AA2024-T3 aluminum alloy used in this study. To confirm the alloy composition was based on standard specifications, the analysis was conducted at Quality Control and Measurement department. The elemental analysis results are given in Table 3.

Table 3. Chemical composition of AA2024-T3 aluminum alloy determined by OES analysis.

Element	Standard composition (wt.%)	Experimental result (wt.%)
Si	Max 0.50	0.0666
Fe	Max 0.50	0.2390
Cu	3.8–4.9	4.00
Mn	0.30–0.90	0.5720
Mg	1.2–1.8	1.33
Zn	Max 0.25	0.1090
Cr	Max 0.10	0.0061
Ni	—	—
Ti	Max 0.15	0.0303
Ga	—	—
V	—	—
Each other element	Max 0.05	0.0107
Total other elements	Max 0.15	0.0107
Al	Balance	93.63

3.4 Fourier Transform Infrared Analysis (FTIR)

Fourier transform infrared spectroscopy (FTIR) used to analyze the functional groups present in Ziziphus leaves powder. The FTIR spectrum (Figure 9), revealed the presence of many bands corresponding to the bioactive phytochemicals like phenolics, flavonoids, tannins, saponins, glycosides, and terpenoid-related group.

The observed bands at 2917–2849 cm^{-1} are assigned to aliphatic C–H stretching vibrations, whereas the band at 1730 cm^{-1} can be assigned to C=O stretching of esters or carboxylics. The band appearing at 1605 cm^{-1} resulting from vibrations of the C=C aromatic chromophore suggests the presence of flavonoids and phenolic compounds. The bands at 1516–1462 cm^{-1} and 1367–1315 cm^{-1} are due to vibrations of aromatic and phenolic groups. The C–O stretching vibrations at 1237, 1161, 1100 and 1009 cm^{-1} indicate the existence of sugar, glycoside and saponin-related compounds. The bands at 764–720 cm^{-1} may be due to substituted aromatic rings.

The presence of functional groups suggests that there are phytochemical constituents that can adsorb on the surface of AA2024-T3 aluminum alloy. This contributes to the formation of a protective layer which then enhances corrosion inhibition performance.

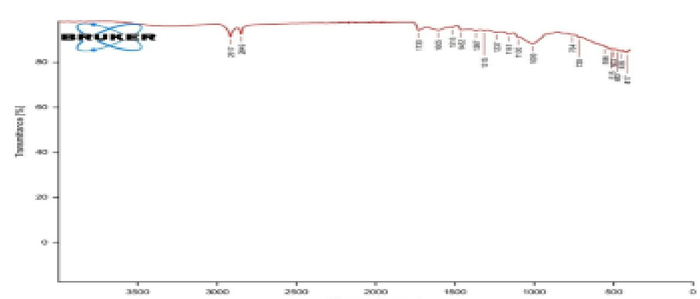


Figure 9. FTIR spectrum of Ziziphus leaves powder showing the characteristic absorption bands of the main functional groups.

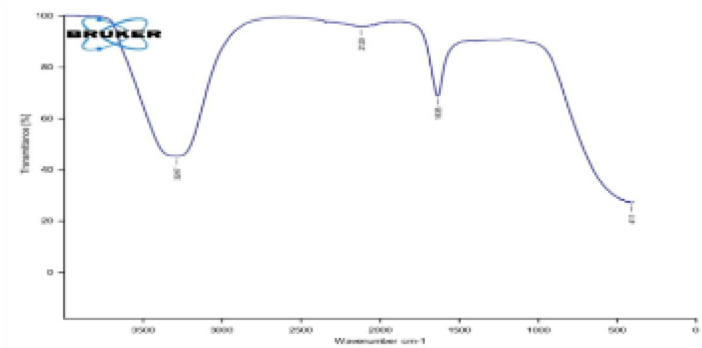


Figure 10. FTIR spectrum of Ziziphus leaves extract showing the characteristic absorption bands of the main phytochemical functional groups.

The FTIR spectrum of Ziziphus leaves extract shown in Figure 10 reveals several characteristic absorption bands corresponding to important phytochemical constituents. The broad band at 3287 cm⁻¹ is attributed to O–H stretching vibrations, indicating the presence of phenolic compounds, flavonoids, and alcohols. The band at 2120 cm⁻¹ may suggest the presence of nitrogen-containing compounds, while the peak at 1635 cm⁻¹ is associated with aromatic and carbonyl-related vibrations, indicating the presence of flavonoids, tannins, and oxidized phenolic compounds. The band at 411 cm⁻¹ may be related to mineral-associated vibrations. These functional groups support the ability of the extract constituents to adsorb onto the alloy surface and form a protective layer against corrosion.

3.5 Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) was used to examine the surface morphology of AA2024-T3 aluminum alloy before and after immersion in 1 M HNO_3, with and without Ziziphus leaves extract. As shown in Figure 11a, the alloy surface before immersion appears relatively smooth, with only slight polishing marks. In the presence of the inhibitor (Figure 11b), the alloy surface is comparatively less damaged and appears to be covered by a protective layer. In contrast, the surface exposed to the acidic solution without inhibitor (Figure 11c) exhibits clear signs of corrosion damage, including pits and surface roughness. These observations confirm the protective effect of Ziziphus leaves extract on the alloy surface.

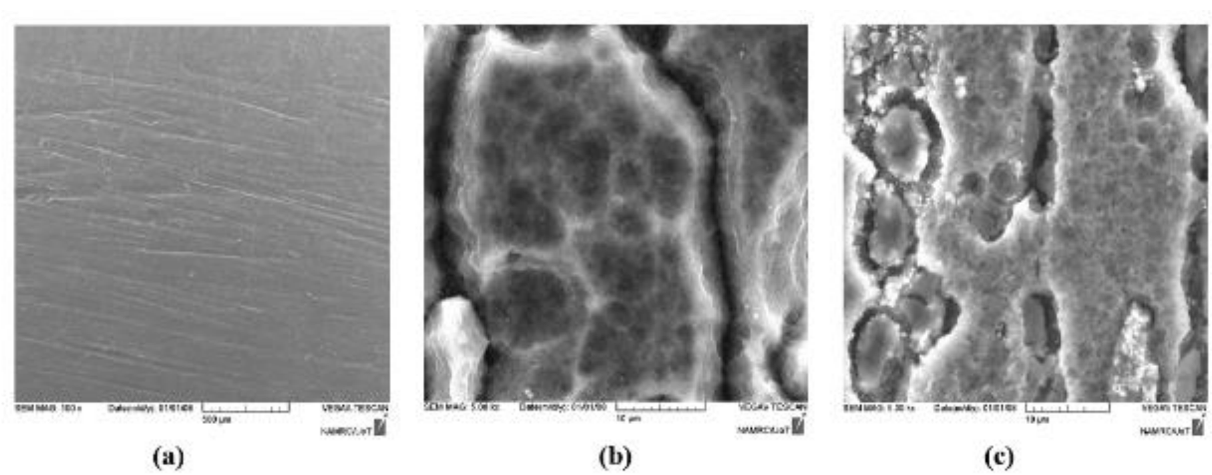


Figure 11. SEM micrographs of AA2024-T3 aluminum alloy surface under different conditions: (a) before immersion, (b) after immersion in 1 M HNO_3 in the presence of Ziziphus leaves extract, and (c) after immersion in 1 M HNO_3 without inhibitor.

4. Conclusion

The present study evaluated the corrosion inhibition performance of Ziziphus leaves extract for AA2024-T3 aluminum alloy in 1 M HNO_3 by the weight loss technique. The obtained results showed that the extract acts as an effective green inhibitor, reducing the corrosion rate significantly under the studied conditions.

It was found that the corrosion rate increased with increasing temperature and decreased with increasing inhibitor concentration. The maximum inhibition efficiency is 92.83% at 40°C and at maximum inhibitor concentration, which showed a very strong protective performance of the extract, in an acidic medium. In the spectra of FTIR functional groups essential phytochemicals were present and with SEM study a protective layer adsorbed on the surface of the alloy was formed. The results of current investigation support the constituents of extract in performing anticorrosion through adsorption at surface.

On the basis of those results, Ziziphus leaves extract may be regarded as a promising and eco-friendly corrosion inhibitor for the AA2024-T3 aluminum alloy in a nitric acid solution.

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