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Electrochemical analysis of 5-Nitro-2-furaldehyde semicarbazone as a mild steel corrosion inhibitor in corrosive solution: An EIS, adsorption and SEM study

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ABSTRACT

The results obtained from EIS, weight loss measurements, adsorption isotherms, and SEM were used to investigate the electrochemical behavior of NFS as a corrosion inhibitor for mild steel in 1.0 M HCl. The inhibition efficiency of NFS the electrode reaction was found to increase with NFS concentration up to a maximum value of 0.5 mM with 92.1 % inhibition efficiency, based on mass loss measurements. The findings revealed that the inhibitive performance of the investigated inhibitor was slightly higher at higher temperatures, indicating improved better protection at elevated thermal conditions. Based on the analysis of the adsorption isotherm studies on mild steel, the findings revealed that the adsorption of NFS can be follows the Langmuir isotherm model, although both chemisorption and physisorption mechanisms were observed. Analysis of the surface morphology by SEM also showed a smooth and resistant surface with less corrosion when NFS is present consistent with previous findings of NFS as a corrosion inhibitor. The inhibition mechanism was further explained in more detail and has given a good understanding regarding the behavior of NFS molecules and steel surface. Therefore, the results demonstrated in this work show that NFS has the potential for acting as a protective barrier against the steel corrosion in corrosive solution.

1. Introduction

Corrosion is a significant challenge in many industrial applications, especially when using cost-effective materials such as mild steel harsh conditions, including acidic environments [1]. Acidic media are commonly encountered in industrial processes like in the oil and gas industries, chemical, mining etc. require the application of acids such as hydrochloric acid (HCl) and sulfuric acid (H $_2$ SO $_4$) in activities like pickling, cleaning, descaling of metals among others. These acids, while essential for industrial applications, are however highly corrosive to metals including mild steel [2]. Consequently, the degradation of steel under such conditions leads to material loss and increased maintenance costs and costs of maintenance, thus reducing industrial output and, in effect, the economy [3]. In order to overcome this problem, corrosion inhibitors are typically used in order to safeguard the formation of metal

surfaces from the damaging impacts of acids. Corrosion inhibitors function by forming a protective barrier to the metal surface which slows down or even inhibits the process of corrosion [4]. There are two main types of corrosion inhibitors: classified into organic and inorganic inhibitors. The inorganic inhibitors, including chromates and phosphates, are widely employed due to their high efficiency in overcoming corrosion. However, they suffer significant environmental concerns as well as toxicity. Inorganic inhibitors produce hazardous by-products and are non-biodegradable leading to strict industrial regulations [5].

On the other hand, organic corrosion inhibitors with nitrogen, oxygen and sulfur heteroatoms being some of the more preferred ones are gaining attention due to their eco-friendly properties due to their benefits in environmental issues. Organic inhibitors are generally soluble in water and highly biodegradable as compared to inorganic inhibitors [6]. Their inhibitive performance is attributed to their functional groups

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which enable them to physisorption through lone pair electrons of electrons and π -electrons to form a layer that reduces corrosive attack [7]. However, inorganic inhibitors often exhibit superior efficiency at higher temperatures or in corrosive settings [8,9]. Among the promising organic inhibitors that have been identified, NFS has demonstrated exceptional potential, this is because that the NFS molecule possesses at the two poles both a nitro group and a semicarbazone grouping. These functional groups improve its propensity to adsorb on metal surfaces so as to mitigate this problem. The nitro group facilitates electron donation the the formation of a protective inhibitor layer [10]. It has been established that semicarbazones demonstrate strong corrosion inhibition properties against corrosion wherein physisorption and chemisorption processes are dominant [11]. This dual adsorption mechanism provides a stable interaction with the metal surface and as such NFS is very suitable for use in corrosive high acidic services [12]. Among the most effective techniques for assessing corrosion inhibition is EIS in order to assess the efficiency of corrosion inhibitors. EIS stands for electrochemical impedance spectroscopy, which provides valuable electrochemical parameters such as charge transfer resistance and double layer capacitance that helps in measuring the inhibition efficiency as well as in finding the nature of the inhibitor [13]. In addition to EIS, adsorption studies utilizing that involve adsorption studies employing techniques such as the Langmuir isotherm which give information regarding the extent and strength of the interaction between the inhibitor and the metal surface [14].

The increasing demand for environmentally friendly corrosion inhibitors has led to a shift toward organic-based compounds that minimize ecological harm while maintaining high inhibition efficiency. In this study, 5-Nitro-2-furaldehyde Semicarbazone (NFS) is explored as a potential green corrosion inhibitor due to its organic nature, metal-free composition, and lower toxicity compared to conventional inhibitors. Unlike traditional corrosion inhibitors that often contain toxic heavy metals such as chromium-based compounds, NFS is an organic molecule with functional groups that promote strong adsorption onto metal surfaces without contributing to environmental contamination. The presence of heteroatoms such as nitrogen and oxygen enhances its inhibition efficiency while reducing the potential for hazardous metal ion release. Additionally, organic corrosion inhibitors, particularly those containing furan and semicarbazone moieties, tend to exhibit higher biodegradability, reducing long-term accumulation in the environment. Many synthetic inhibitors, including chromates and phosphonates, have been restricted due to their toxicity and persistence in water systems. In contrast, NFS provides an alternative with lower toxicity, making it a safer choice for industrial applications where metal cleaning and acid treatments are required. Moreover, the adsorption strength of NFS onto the mild steel surface minimizes its desorption into industrial wastewater, thereby reducing its potential impact on aquatic environments. Future studies focusing on its biodegradability profile, eco-toxicity assessment, and green synthesis optimization could further enhance its application as a sustainable and environmentally friendly corrosion inhibitor.

In the present investigation, the result of the barrier property of NFS (Fig. 1) as a corrosion inhibitor for mild steel in an acidic environment by EIS, adsorption isotherm and SEM. This study presents the first

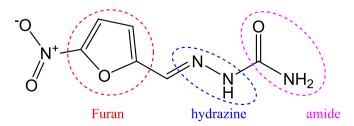


Fig. 1. Molecular structure of 5-Nitro-2-furaldehyde Semicarbazone (NFS) highlighting the functional groups: furan, hydrazine, and amide moieties.

electrochemical investigation of 5-Nitro-2-furaldehyde Semicarbazone (NFS) as a corrosion inhibitor for mild steel in acidic environments, utilizing a comprehensive approach combining Electrochemical Impedance Spectroscopy (EIS), adsorption isotherms, and Scanning Electron Microscopy (SEM). Unlike previously studied inhibitors, NFS contains a unique combination of furan, semicarbazone, and nitro functional groups, which contribute to enhanced adsorption on the metal surface through both physisorption and chemisorption mechanisms. Moreover, the study provides a detailed thermodynamic and mechanistic analysis, including the Langmuir adsorption model, Gibbs free energy calculations, and temperature-dependent inhibition performance, which further validates the effectiveness of NFS. The dual nature of adsorption, along with the formation of a stable inhibitor-metal complex, distinguishes NFS from conventional inhibitors. Additionally, this research explores the impact of temperature variations and competitive adsorption with chloride ions, offering new insights into the stability and efficiency of NFS under industrially relevant conditions. These findings highlight NFS as a promising, efficient, and potentially sustainable alternative for corrosion protection in acidic environments, contributing to advancements in green and high-performance corrosion inhibitors. The primary objectives of this research are to evaluate the inhibition efficiency of NFS at different concentrations, analyze the effect of temperature on the corrosion inhibition process and elucidate the adsorption mechanism. This study aims to contribute to the existing body of knowledge on NFS as a cost-effective, highly efficient, and environmentally friendly corrosion inhibitor, which is a cost-effective, efficient and eco-friendly corrosion inhibitor particularly for industrial applications in acidic environments. By integrating EIS to assess electrochemical parameters weight loss measurements to determine longterm inhibition efficiency, and SEM for surface morphology analysis this research provides a comprehensive evaluation of the corrosion protection properties of NFS.

2. Experimental

2.1. Materials

Mild steel specimens were used for the corrosion inhibition experiments, with the following chemical composition: Mild steel specimens were used for the corrosion inhibition experiments, with the following chemical composition: Fe (99. 21 %), carbon (0. 21 %), silicon (0.38 %), phosphorous (0. 09 %), sulfur (0.05 %), manganese (0. 05 %) and aluminum (0.01 %). The specimens were prepared based on ASTM G1-03 standard [15,16] involving mechanical polishing using emery paper of different grit sizes 400, 600 and 1000 to achieve a uniform surface finish. After polishing, the specimens were further polished with 600-grit SiC paper and followed by sequential cleaning with acetone and distilled water to remove residual grease or contaminants Finally, the specimens were air-dried to prevent oxidation before use. This process ensures uniform surface conditions across all experiments. The corrosive medium utilized in this study was 1.0 M hydrochloric acid (HCl), prepared using analytical-grade reagents and distilled water to ensure high experimental accuracy and reproducibility. The synthesized corrosion inhibitor, 5-Nitro-2-furaldehyde semicarbazone (NFS), was used without further purification to assess its direct applicability. The inhibitor concentrations investigated in this study were 0. 1 mM, 0. 2 mM, 0. 3 mM, 0. 4 mM, and 0. 5 mM. All experiments employed high-purity analytical reagent-grade chemicals, ensuring reliable and reproducible results.

2.2. Electrochemical impedance spectroscopy (EIS)

In addition to Electrochemical Impedance Spectroscopy (EIS), weight loss measurements were also conducted to assess the long-term inhibition efficiency of NFS. While weight loss analysis provides an overall measure of corrosion rate reduction over extended immersion

periods, EIS was specifically selected to evaluate the inhibitor's real-time electrochemical behavior, particularly its influence on charge transfer resistance (R_{ct}) and double-layer capacitance (C_{dl}). This combination allows for a comprehensive understanding of both adsorption characteristics and the protective film formation on the metal surface. Although potentiodynamic polarization and cyclic voltammetry could offer additional electrochemical insights, our focus on adsorption behavior and inhibitor-film stability justified the use of EIS alongside weight loss techniques.

EIS is an electrochemical technique that uses a small AC potential to determine the impedance response of a system, which gives information on various electrochemical processes that may be occurring at the metal-electrolyte interface. This non-destructive, non-contact method and allows for the evaluation of critical parameters such as charge transfer resistance, the double-layer capacitance, diffusion characteristics and efficiency of the corrosion inhibitors [17,18]. The Nyquist plot is the most widely used graphical representation of EIS data generated in any corrosion studies. Nyquist plot plots imaginary impedance (Zimag) on the vertical axis against real impedance (Z_{real}) on the horizontal axis. Typically, Nyquist plots display a semicircular arc in the Nyquist plot and the major diameter of the arc is the R_{ct}. A larger semicircle signifies a higher Rct, which correlates with improved corrosion resistance due to the formation of a protective inhibitor layer. The high-frequency region of the Nyquist plot provides information about solution resistance (R_s), while the low-frequency region reflects the charge transfer process at the metal-electrolyte interface [19,20]. In real electrochemical systems, Nyquist plots often exhibit depressed semicircles or flattened arcs instead of ideal semicircles. These deviations are primarily attributed to surface roughness, inhomogeneities, or non-uniform adsorption of the inhibitor. To account for these non-ideal behaviors, a constant phase element (CPE) is introduced into the equivalent circuit model to provide a more accurate representation of the electrochemical system.

EIS was employed to assess the corrosion inhibition efficiency of NFS for mild steel in 1.0 M HCl. All EIS experiments were conducted using a three-electrode system configuration. Mild steel served as the working electrode, platinum as the counter electrode, and a saturated calomel electrode (SCE) as the reference electrode. The working electrode, with an exposed surface area of 1 cm², was immersed in an acidic solution containing different concentrations of NFS inhibitor. The experiments were carried out at various NFS concentrations; 0.1 mM, 0. 2 mM, 0. 3 mM, 0. 4 mM and 0. 5 mM to determine the optimal concentration for effective corrosion inhibition. Electrochemical impedance spectroscopy measurements were conducted with a Gamry Potentiostat/ Galvanostat/ ZRA model Ref 600 with interfacing by the DC 105 and EIS300 firmware. All electrochemical experiments were conducted at room temperature (~298 K). Impedance measurements were carried out at the OCP after the stabilization of the system with an AC amplitude of 5 mV peak-to-peak superimposed on the steady-state potential. The frequency range for impedance measurements spanned from 0.1 Hz to 100 kHz, ensuring a comprehensive analysis of electrochemical behavior. To ensure the reliability of our results, all experiments were performed in triplicate

2.3. Data analysis and calculation of inhibition efficiency

The impedance data were further analyzed to determine the R_{ct} , which provides valuable insights into the corrosion inhibition performance of NFS [21,22]. The inhibition efficiency (IE%) of NFS was calculated based on the R_{ct} values using the following eq. (1),

$$\label{eq:energy} \textit{IE}\% = \frac{R_{ct(inh)} - R_{ct(uninh)}}{R_{ct(inh)}} \times 100 \tag{1}$$

where: $R_{ct(inh)}$ is the charge transfer resistance in the presence of the inhibitor, and $R_{ct(uninh)}$ is the charge transfer resistance in the absence of the inhibitor.

Higher R_{ct} values in the presence of NFS indicate enhanced corrosion resistance due to the formation of a protective inhibitor layer on the mild steel surface, effectively reducing charge transfer at the metal-electrolyte interface. The increase in IE% with inhibitor concentration further confirms the strong adsorption of NFS molecules, leading to improved surface protection.

2.4. Weight loss measurements

Weight loss experiments were conducted over a 5-h immersion period, at temperatures ranging from 303 K to 333 K. Mild steel coupons were exposed to various inhibitor concentrations of 0, 0.1, 0.2, 0.3, 0.4, 0.5, and 1 mM. After exposure, the mild steel specimens were thoroughly thoroughly cleaned with distilled water and acetone, dried in an oven, and weighed. To ensure accuracy and reproducibility, each experiment was performed in triplicate, and the mean weight loss values were calculated [23,24]. The corrosion rate (C_R) and inhibition efficiency (IE) were determined using Eqs. (2) and (3):

$$C_R = \frac{87600 \times W}{a \times t} \tag{2}$$

where W represents the weight loss of the tested mild steel (grams), a denotes the surface area of the tested mild steel (cm²), and t represents the exposure time in hours.

$$IE\% = \frac{C_{R(o)} - C_{R(i)}}{C_{R(o)}} \times 100$$
 (3)

where $C_{R(o)}$ represents the corrosion rate in the absence of the inhibitor, and $C_{R(i)}$ represents the corrosion rate in the presence of the inhibitor.

2.5. Scanning electron microscopy

SEM analysis was conducted to examine the surface morphology of mild steel specimens before and after immersion in 1.0 M HCl. The specimens were immersed in 1.0 M HCl solution, both with and without the inhibitor and without the inhibitor. The specimens were immersed at 303 K for 5 h and after which they were washed with distilled water, dried, and prepared for SEM imaging. A high-resolution scanning electron microscope was utilized to evaluate surface degradation, corrosion-induced damage, and the protective barrier formed by the inhibitor [25,26].

3. Results and discussion

3.1. Open circuit potential (OCP) variation over time

The OCP measurements provide valuable insights into the electrochemical stability of mild steel in 1.0 M HCl with and without NFS as a corrosion inhibitor. The variation of OCP over time was recorded to ensure system stabilization before conducting EIS. Initially, the blank solution (uninhibited 1.0 M HCl) exhibited the most negative OCP value (Fig. 2), indicating the high susceptibility of mild steel to corrosion in the acidic medium. However, with the addition of NFS, the OCP values shifted toward more positive potentials, with the 0.5 mM NFS concentration showing the highest initial shift to -410 mV vs. SCE. This shift suggests that the adsorption of NFS molecules onto the mild steel surface begins almost immediately upon immersion, forming a protective layer that reduces metal dissolution. Over time, the OCP values gradually stabilized, reaching a steady-state equilibrium within approximately 10-15 min. The stabilization of OCP confirms that a balance between inhibitor adsorption and desorption is achieved, ensuring that the metal surface is sufficiently protected before EIS measurements. The final OCP values after 30 min further reinforce this trend, with the blank solution maintaining an OCP of -450 mV vs. SCE, whereas the system containing 0.5 mM NFS stabilized at -404 mV vs. SCE. The shift in OCP with

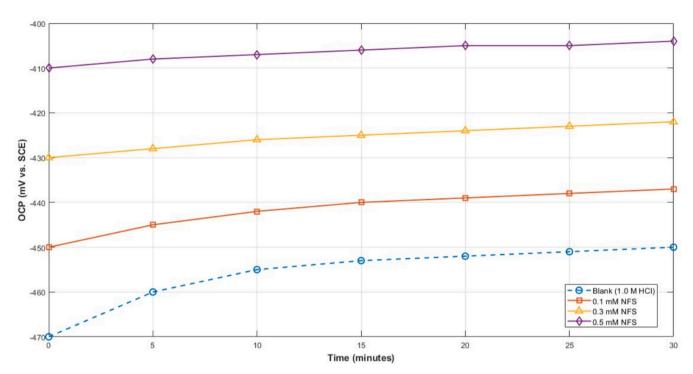


Fig. 2. Open Circuit Potential OCP Variation Over Time for Mild Steel in 1.0 M HCl with Different NFS Concentrations

increasing NFS concentration suggests that the inhibitor effectively suppresses anodic dissolution by reducing the reactivity of the steel surface. The progressive increase in OCP with increasing NFS concentration supports the mixed-type inhibition mechanism of NFS, as it influences both anodic and cathodic reactions. The observed positive shift in OCP confirms that NFS successfully mitigates corrosion by forming a stable protective layer on the mild steel surface. These results validate the effectiveness of NFS as a corrosion inhibitor and confirm that the system reached electrochemical stability before conducting further

impedance measurements.

3.2. Equivalent circuit model

For the analysis of EIS data, an equivalent electrical circuit model (Fig. 3a) has been used which includes the R_{s} , R_{ct} , and a finite CPE representing the double layer capacitance The CPE was incorporated to account for non-ideal electrochemical behavior arising from surface roughness or electrode heterogeneity due to surface roughness or

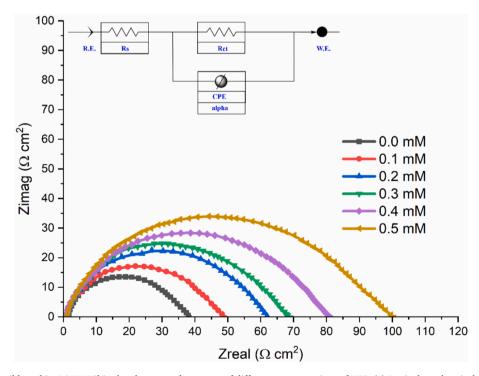


Fig. 3. Nyquist plots for mild steel in 1.0 M HCl in the absence and presence of different concentrations of NFS. (a) Equivalent electrical circuit model; (b) Nyquist plots at varying inhibitor concentrations.

heterogeneity at the electrode interface. This model enables the accurate determination of R_{ct} where higher R_{ct} values correspond to lower corrosion rates [27,28].

3.3. Electrochemical impedance spectroscopy (EIS) analysis

The electrochemical behavior of NFS was analyzed using both EIS and weight loss methods to ensure a comprehensive evaluation of its inhibition efficiency. EIS provides real-time insights into charge transfer resistance and adsorption characteristics, while weight loss analysis complements this by quantifying long-term corrosion rate reduction. The results from both techniques show a strong correlation, with increasing NFS concentration leading to higher inhibition efficiency. The adsorption behavior observed in EIS measurements aligns well with the adsorption isotherm derived from weight loss data, reinforcing the effectiveness of NFS as a corrosion inhibitor.

The Nyquist plots (Fig. 3b) illustrate the electrochemical impedance spectra for mild steel in 1.0 M HCl, with varying concentrations of NFS. The effect of NFS on corrosion resistance was evaluated at concentrations of 0.0, 0.1, 0.2, 0.3, 0.4, and 0.5 mM. on the corrosion resistance is obtained from 0.0, 0.1, 0.2, 0.3, 0.4 and 0.5 mM NFS. The semicircular nature of the impedance spectra indicates that the corrosion process is predominantly controlled by charge transfer. Thus, the progressive increase in the semicircle diameter with higher NFS concentrations confirms the enhanced corrosion protection provided by the inhibitor. [29]. Table 1 summarizes the electrochemical impedance parameters for mild steel in 1.0 M HCl at different NFS concentrations. The principal parameters are $R_{\rm Ct}$, Rs, $C_{\rm dl}$ and inhibition efficiency (IE%). From Table 1, we can draw the following conclusions:

- 1. It shows that R_{ct} increases with the increase of NFS concentration because, NFS has been able to coordinate the electron transfer between the two electrodes. This shows that the inhibitor is able to form a layer on the surface of the mild steel through which the corrosion cannot penetrate hence increasing the resistance to corrosion.
- 2. As in Fig. 3, with the increase of NFS concentration, the value of C_{dl} reduces, which Confirms the formation of a protective layer on the steel surface. The C_{dl} values obtained are lower due to which fewer active sites are exposed for the corrosive attack due to the adsorption of NFS molecules. This can be observed in the fact that C_{dl} reduced sharply from 585. 8 μ F \bullet cm 2 (uninhibited) to 110.2 μ F \bullet cm 2 (0.5 mM NFS).
- 3. The inhibition efficiency increases proportionally with NFS concentration, reaching a maximum of 62.3 % at 0.5 mM. This proves that NFS effectively protects against corrosion and as concentration increases it approaches the saturation level where inhibitor is presumably coating the surface in maximum degree.
- R_s remains nearly constant across different NFS concentrations, indicating that the inhibitor does not significantly affect the solution conductivity.

Table 1Electrochemical Impedance Parameters for Mild Steel in 1.0 M HCl with Different Concentrations of NFS.

Conc. (mM)	$R_{\rm ct}$ ohm·cm ²	$R_{\rm s}$ ohm·cm ²	Yo (CPEdl) ($\times 10^6$ s ⁿ · Ω^{-1} ·cm ⁻²)	IE%
0	38	1.47	585.8	_
0.1	48	1.59	325.2	20.8
0.2	63	1.64	298.2	39.6
0.3	69	1.69	215.7	44.9
0.4	82	1.74	187.3	53.6
0.5	101	1.79	110.2	62.3

3.4. Effect of NFS concentration:

The electrochemical impedance parameters, which include $R_{ct},\,R_s,\,C_{dl},$ and inhibition efficiency (IE%), are summarized in Table 1. The results show a significant increase in R_{ct} values with increasing NFS concentration, at the same time as the C_{dl} values lower, suggesting the formation of a defensive layer at the slight steel surface [30,31]. The R_{ct} value is 42.3 $\Omega \bullet cm^2$, indicating rapid corrosion in the absence of the inhibitor. The high C_{dl} value (585.8 $\mu F \bullet cm^2$) suggests a large area of exposed steel surface, leading to high corrosion rates.

- At 0.1 mM NFS: The addition of NFS causes a sharp increase in R_{ct} with a corresponding decrease in C_{dl} to 325.8 $\mu F \bullet cm^2$. The inhibition efficiency (IE%) is 20.8 %, indicating a considerable reduction in the corrosion rate.
- At 0.2 mM NFS: R_{ct} further increases to 63 Ω •cm², and C_{dl} decreases to 298.2 μ F•cm², with an IE% of 39.6 %. This improvement is attributed to the adsorption of more NFS molecules on the metal surface
- At 0.3 mM NFS: The R_{ct} value reaches 69 Ω•cm², and C_{dl} reduces to 215.7 μF•cm², corresponding to an IE% of 44.9 %. The continued increase in R_{ct} indicates the growth of the protective layer.
- At 0.4 mM NFS: The R_{ct} increases to 82 Ω•cm², with a further reduction in C_{dl} to 187.3 μF•cm². The inhibition efficiency improves to 53.6 %, indicating substantial surface coverage by the inhibitor.
- At 0.5 mM NFS: The highest R_{ct} value of 101 Ω•cm² is achieved, with the lowest C_{dl} value of 110.2 μF•cm². The inhibition efficiency reaches a maximum of 62.3 %, showing that the surface is wellprotected by the adsorbed NFS molecules.

Nyquist plots (Fig. 3b) provide a visual representation of the electrochemical impedance spectra by showing the increasing diameter of the semicircles as the concentration of NFS increases. Each plot corresponds to a specific NFS concentration, and the observed increase in semicircle diameter with increasing NFS concentration confirms the enhanced corrosion protection. The semicircles become larger and more pronounced as NFS concentration increases from 0.1 mM to 0.5 mM, reflecting the enhanced protection provided by the inhibitor [32,33]. The Nyquist plots also show that the corrosion process is predominantly charge transfer-controlled, as evidenced by the near-perfect semicircles. This suggests that the corrosion inhibition mechanism is generally due to the adsorption of NFS onto the slight steel surface, which impedes electron transfer between the metal and the corrosive medium [34]. The combined data from Table 1 and Fig. 3 strongly support the conclusion that NFS significantly enhances corrosion resistance for moderate metallic in 0.1 M HCl. As the concentration of NFS will increase, Rct will increase extensively, and Cdl decreases, indicating the formation of a defensive layer at the metallic surface. The inhibition efficiency (IE%) reaches a maximum of 62.3 % at 0.5 mM NFS, displaying that NFS offers notable safety toward corrosion. The regular behavior in both the impedance data and the Nyquist plots confirms the robust adsorption of NFS molecules, which block the lively corrosion sites and slow down the corrosion manner [35].

3.5. Effect of temperature on corrosion rate and inhibition efficiency

The effect of temperature on the corrosion rate (CR) of moderate metallic in 1 M hydrochloric acid and the inhibition performance (IE%) of 5-Nitro-2-furaldehyde semicarbazone (NFS) was investigated the use of the weight loss technique. Experiments were conducted at temperatures ranging from 303 K to 333 K to evaluate the influence of thermal variations on both corrosion rate and inhibitor performance. The data, illustrated in Fig. 4, reveal two key trends, the corrosion rate decreases with increasing inhibitor attention, and the inhibition performance improves barely because the temperature increases.

The C_R in $mg.cm^2.h^{-1}$ is plotted in opposition to C_{inh} at different

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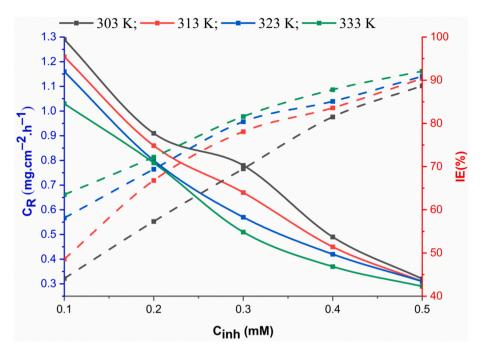


Fig. 4. Effect of NFS concentration on corrosion rate (CR) and inhibition efficiency (IE%) for mild steel in 1.0 M HCl at different temperatures (303 K, 313 K, 323 K, and 333 K).

temperatures in Fig. 4 (strong lines). At every temperature, the corrosion charge decreases with increasing NFS efficiency, indicating that NFS efficaciously reduces the rate of steel dissolution in acidic environments. However, it's miles vital to note that at higher temperatures (e.g., 333 K), the corrosion charge in the absence of the inhibitor is decreased than at decrease temperatures (e.g., 303 K). This may be attributed to the thermally activated adsorption of NFS molecules onto the slight metallic surface, imparting more green protection at accelerated temperatures. At 303 K, the corrosion rate decreases from 1.29 mg.cm².h $^{-1}$ at 0.1 mM NFS to 0.32 mg.cm 2 .h $^{-1}$ at 0.5 mM NFS. At 333 K, the corrosion rate shows a similar trend, decreasing from 1.03 mg.cm 2 .h $^{-1}$ at 0.1 mM to 0.29 mg.cm 2 .h $^{-1}$ at 0.5 mM, indicating sturdy corrosion inhibition even at higher temperatures. These results confirm that NFS remains highly effective across a wide temperature range, demonstrating strong performance at both ambient and elevated temperatures [36,37].

The inhibition efficiency (IE%), plotted in Fig. 4 as dashed traces, increases barely with rising temperature in any respect NFS concentrations. The development in inhibition performance with temperature shows that the adsorption of NFS molecules onto the metallic surface is a thermally activated process, that is likely a combination of physisorption and chemisorption mechanisms. At 303 K, the inhibition performance is 88.7 % at 0.5 mM NFS, at the same time as at 333 K, the efficiency increases barely to 92.1 %, demonstrating better protecting behavior at higher temperatures. Similarly, at lower concentrations (0.1 mM), the inhibition performance improves from 44.1 % at 303 K to 63.5 % at 333 K, suggesting that even at low concentrations, NFS can provide enormous protection at expanded temperatures.

The mild increase in inhibition performance with temperature shows that NFS adsorption onto the slight metallic surface is facilitated via both physisorption and chemisorption. At higher temperatures, the expanded kinetic power of the device complements the adsorption rate of NFS molecules, leading to progressed surface insurance [38]. The thermodynamic parameters, which includes the free strength of adsorption, in addition affirm that NFS adsorption follows a combined adsorption mechanism. The discount in CR values with increasing temperature, as located inside the weight reduction information, supports the conclusion that the inhibitor paperwork a stronger and extra stable protecting layer at expanded temperatures, correctly decreasing

the energetic surface vicinity uncovered to the corrosive medium [39]. The effect of temperature on the corrosion inhibition performance of NFS indicates that the inhibitor remains highly effective across the temperature range of 303–333 K. As the temperature increases, both the corrosion rate decreases and the inhibition efficiency improves slightly, suggesting that NFS exhibits strong protective behavior in acidic environments even at elevated temperatures. The results highlight the NFS potential to be used as a inhibitor for corrosion in industrial process where varying thermal conditions are common.

It is noted that inhibition efficiency (IE%) values obtained from EIS and weight loss measurements exhibit some variation, with weight loss generally yielding higher efficiency values. This discrepancy arises because weight loss provides an average corrosion rate reduction over an extended immersion period, whereas EIS captures instantaneous electrochemical behavior at a given time point. The difference is expected, as weight loss measurements allow for prolonged inhibitor adsorption and stabilization, leading to more complete surface coverage over time. Despite these differences, both methods confirm the strong protective effect of NFS on mild steel in 1.0 M HCl, supporting the proposed adsorption mechanism.

3.6. Adsorption isotherm analysis:

In this study, weight loss measurements were primarily used to evaluate the adsorption isotherms of 5-Nitro-2-furaldehyde Semicarbazone (NFS) due to their ability to provide a direct assessment of the inhibitor's long-term effectiveness. However, Electrochemical Impedance Spectroscopy (EIS) was also employed as a complementary technique to further investigate the electrochemical behavior of NFS on the mild steel surface. While weight loss techniques measure the overall corrosion rate reduction due to adsorption, EIS provides real-time insights into $R_{\rm ct}$ and double-layer capacitance ($C_{\rm dl}$), both of which are indicative of surface coverage and protective film formation. The lower $C_{\rm dl}$ values obtained from EIS suggest the displacement of water molecules by adsorbed inhibitor molecules, reinforcing the findings from weight loss measurements. Additionally, EIS supports the differentiation between physisorption and chemisorption mechanisms by analyzing variations in $R_{\rm ct}$ and $C_{\rm dl}$, strengthening the interpretation of the

adsorption isotherms derived from weight loss methods. By integrating EIS with weight loss techniques, this study ensures a comprehensive analysis of the inhibition efficiency and adsorption behavior of NFS, offering both short-term electrochemical insights and long-term corrosion inhibition performance.

The adsorption behavior of NFS on the mild steel surface was analyzed using Langmuir and Temkin adsorption isotherms to determine the most suitable model describing the interaction between the inhibitor molecules and the metal surface. The surface coverage (θ) was calculated from weight loss measurements and plotted against the Cinh to evaluate the adsorption mechanism. The best-fitting isotherm was determined based on the correlation coefficient (R2), which served as the primary criterion for selection. The Langmuir adsorption isotherm, which assumes monolayer adsorption on a homogeneous surface without interactions between adsorbed molecules, provided the best correlation with the experimental data, exhibiting R² values close to unity. This suggests that NFS molecules uniformly adsorb on active sites of the mild steel surface, forming a protective film that significantly reduces corrosion. On the other hand, the Temkin adsorption isotherm, which considers interactions between adsorbed molecules, showed a slightly lower correlation, indicating that NFS adsorption is predominantly governed by Langmuir behavior rather than a multi-layered or interaction-dependent mechanism [40,41]. These results confirm that the adsorption of NFS on mild steel in acidic media is spontaneous, stable, and primarily follows a monolayer adsorption model, supporting the proposed mixed physisorption and chemisorption inhibition mechanism. The obtained data were fitted to different adsorption models, and the best correlation was found with Langmuir's adsorption isotherm, as depicted in Fig. 5.

Langmuir's adsorption isotherm is mathematically expressed as in eq. (4):

$$\frac{C_{inh}}{\theta} = \frac{1}{K} + C_{inh} \tag{4}$$

where: C_{inh} is the concentration of the inhibitor.

K is the equilibrium constant of the adsorption process, which is related to the strength of the interaction between the inhibitor molecules and the metal surface.

In Fig. 5, the plot of $C_{inh} \setminus \theta$ versus C_{inh} produces a straight line, which confirms that the adsorption of NFS on the mild steel surface follows Langmuir's isotherm. The slope and intercept of the line were used to determine the equilibrium constant (K), and the intercept values were used to calculate the standard free energy of adsorption (ΔG_{ads}).

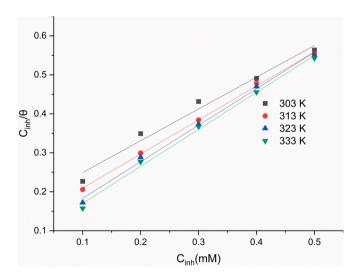


Fig. 5. Langmuir adsorption isotherms for the adsorption of NFS on mild steel at different temperatures (303 K, 313 K, 323 K, and 333 K).

3.7. Thermodynamic parameters of adsorption

The standard free energy of adsorption (ΔG_{ads}) was calculated [61,62] using the following eq. (5):

$$K = \frac{1}{55.5} exp \frac{-\Delta G_{ads}^o}{RT} \tag{5}$$

where R is the universal gas constant (8.314 $J \cdot mol^{-1} \cdot K^{-1}$), T is the absolute temperature in Kelvin, and 55.5 is the molar concentration of water in the solution.

The negative values of ΔG_{ads} , presented in Table 2, confirm that the adsorption process is spontaneous, and that the adsorbed layer remains stable on the mild steel surface. The increasing negativity of ΔG_{ads} with temperature indicates that the adsorption of NFS becomes more thermodynamically favorable at elevated temperatures. For instance, at 303 K, $\Delta G_{ads} = -35.56$ kJ/mol, while at 333 K, $\Delta G_{ads} = -41.67$ kJ/mol, demonstrating an increasing tendency toward stronger adsorption interactions.

The magnitude of ΔG_{ads} provides insight into the nature of adsorption. Typically, ΔG_{ads} values around -20~kJ/mol or higher (less negative) suggest physisorption, whereas values near -40~kJ/mol or lower (more negative) indicate chemisorption. The ΔG_{ads} values for NFS at all studied temperatures are close to -40~kJ/mol, suggesting that chemisorption is the dominant adsorption mechanism, with physisorption contributing at lower temperatures [41].

3.8. Adsorption mechanism

The chemisorption of NFS on the mild steel surface involves the formation of strong chemical bonds between the inhibitor molecules and metal atoms. The presence of functional groups, such as the nitro (-NO₂) group and semicarbazone (-NH-C(=O)-NH₂) moiety, enhances electron donation to the metal surface, reinforcing the protective effect against corrosion. At lower temperatures, a small contribution from physisorption occurs due to weaker van der Waals interactions; however, chemisorption becomes dominant at elevated temperatures, resulting in higher inhibition efficiency [42]. At lower temperatures, a small contribution from physisorption occurs due to weaker van der Waals interactions; however, chemisorption becomes dominant at elevated temperatures, resulting in higher inhibition efficiency. The process is predominantly chemisorptive, indicating strong molecular interactions between NFS and the mild steel surface, ultimately leading to effective corrosion inhibition.

3.9. Scanning electron microscopy (SEM) analysis

The surface morphology of mild steel immersed in 1.0 M HCl, both in the absence and presence of NFS, was investigated using Scanning Electron Microscopy (SEM). Fig. 6 (a) shows the SEM image of the mild steel surface after immersion in 1.0 M HCl without the inhibitor, while Fig. 6 (b) depicts the surface after immersion in the acid solution containing the inhibitor. In the absence of NFS, the SEM image of the mild steel surface (Fig. 6 a) reveals severe surface degradation [43]. The SEM image clearly illustrates extensive corrosion, characterized by rough, uneven textures with deep pits and cracks. These features are indicative of extensive metal dissolution caused by the aggressive attack of chloride ions in the acidic medium. The corrosive medium promotes intense metal dissolution, resulting in significant structural damage. This morphology is constant with a high corrosion rate, as reflected by the low R_{ct} observed in the EIS measurements. In contrast, Fig. 6(b) demonstrates a smoother and more uniform surface in the presence of NFS. The presence of the inhibitor has significantly mitigated corrosion, leading to the formation of a protective layer on the metal surface. The smoother surface morphology suggests that the inhibitor effectively adsorbed onto the mild steel, creating a barrier that prevents direct

Table 2Thermodynamic parameters for the adsorption of NFS on mild steel at different temperatures.

Temp.	303 K	313 K	323 K	333 K
Intercept	0.167×10^{-3}	0.122×10^{-3}	$0.089 imes 10^{-3}$	0.075×10^{-3}
Slope	0.815	0.873	0.937	0.949
R2	0.979	0.998	0.996	0.995
ΔG_{ads}^{o}	-35.56	-37.12	-37.95	-41.67
Adsorption type	Both physisorption and chemisorption	Both physisorption and chemisorption	Both physisorption and chemisorption	Chemisorption

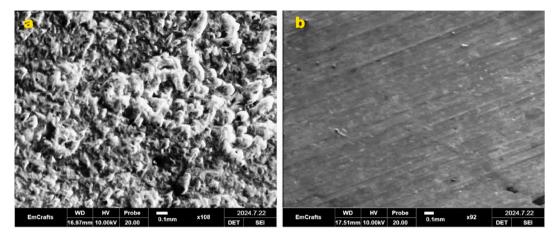


Fig. 6. SEM images of sample surface after exposed to 1.0 M HCl (a) without *inhibitor*, showing severe corrosion damage, and (b) with *inhibitor*, showing a smooth surface protected by the inhibitor.

interaction between the metal and the corrosive medium. This protective film minimizes surface damage and inhibits the metal dissolution process. The development in surface morphology is consistent with the high inhibition efficiency found in the EIS effects, wherein the Rct extended significantly with the addition of the inhibitor. The SEM evaluation supports the realization that NFS acts as a powerful corrosion inhibitor by using forming a solid, protective movie at the slight steel surface, thereby reducing the corrosion rate and preserving the integrity of the metallic. The SEM images provide clear visual evidence of the protective effect of NFS on mild steel in 1.0 M HCl. n the absence of the inhibitor, the metal surface experiences severe corrosion damage, whereas the presence of the inhibitor results in a significantly smoother and less deteriorated surface, confirming its role as an effective corrosion inhibitor. The formation of a protective film on the steel surface is responsible for inhibiting both anodic and cathodic reactions, leading to the observed reduction in the corrosion rate.

3.10. Comparison between NFS and other corrosion inhibitors in acidic solutions

In corrosion inhibition research, various organic inhibitors, particularly those with furan, hydrazine, and amide functionalities, have been widely studied due to their efficiency in protecting metals in acidic media. The inclusion of heteroatoms like nitrogen, sulfur, and oxygen in these compounds facilitates strong adsorption onto metal surfaces through interactions that contribute to forming protective layers, significantly reducing corrosion rates. This study investigates NFS, which has demonstrated promising inhibition efficiency for mild steel in 1.0 M HCl solution. To contextualize its effectiveness, we compare NFS with similar inhibitors.

1. Performance of NFS

NFS achieves an inhibition efficiency of up to 92.1~% at a 0.5~mM concentration in 1.0~M HCl, a performance comparable to or exceeding that of other common inhibitors in similar conditions. NFS operates

effectively at relatively low concentrations and demonstrates stability across a range of temperatures, making it suitable for industrial applications where temperature variability is common [44].

2. Comparative Analysis with Similar Inhibitors

- Furan-2-ylmethylene-Hydrazide (FATH): According to studies, FATH
 achieved an inhibition efficiency of 99.9 % for carbon steel in a 0.1 M
 HCl solution at 600 ppm. FATH's adsorption follows the Langmuir
 isotherm, indicating a robust protective layer formation at higher
 concentrations, similar to NFS's behavior in hydrochloric acid environments [45].
- Furan-based Hydrazine Derivatives: Compounds like (2E)-2-(furan-2-ylmethylidene) hydrazine carbothioamide (FMHC) have shown moderate inhibition, reaching a maximum efficiency of 60 % in 0.5 M H₂SO₄ at 303 K. This compound adheres to the Langmuir adsorption model, similar to NFS, but with lower inhibition performance, which may be attributed to the absence of specific electron-donating groups present in NFS that enhance its adsorption strength [46].
- 4-(2-Mercapto-1,3,4-oxadiazole-5-yl)pyridine (MOP): MOP, another
 organic inhibitor, achieved 93.6 % efficiency for mild steel in 1 M
 HCl at a concentration of 0.5 mM. This efficiency is comparable to
 NFS, though MOP's inhibition mechanism includes substantial surface coverage and electronic interactions that may vary in strength
 based on structural differences, such as the presence of sulfur [47].
- Furfural Derivatives: Compounds like furan-2-carbaldehyde (FF1) and hydroxymethyl furfural have demonstrated efficacy in acid media. These derivatives display promising adsorption behavior on metal surfaces, especially Fe(110), due to favorable electronic and steric factors. Their adsorption energies indicate effective metalinhibitor interaction, aligning with the inhibition mechanism observed for NFS [48].

3. Advantages and Disadvantages

- Advantages of NFS: High inhibition efficiency at low concentrations.
 Dual adsorption mechanism (physisorption and chemisorption)
 providing stable protective layers. Stability at elevated temperatures,
 enhancing applicability in dynamic industrial environments.
- Disadvantages of NFS: Limited testing across non-acidic media, potentially restricting its versatility. Environmental concerns due to the presence of halogenated components, which may affect its suitability in applications requiring eco-friendly solutions.

NFS shows a high inhibition efficiency comparable to effective inhibitors such as MOP and FATH, yet with the potential for optimization in non-acidic environments and environmental impact reduction. Future studies could further explore the synergistic effects of functional groups in NFS, such as the furan ring and hydrazine moiety, to enhance its ecofriendliness and performance across diverse corrosive conditions.

3.11. Comparison with other corrosion inhibitors

Various organic inhibitors, especially those containing furan, hydrazine, or amide functionalities, have been studied for their corrosion inhibition properties in acidic environments. Table 3 provides a comparative overview of several inhibitors with these functional groups, highlighting their inhibition efficiency, metal type, and the type of acidic solution used.

NFS exhibits an excellent inhibition efficiency of 92.1 % at a concentration of 0.5 mM, making it highly competitive with other effective inhibitors containing furan, hydrazine, or amide functionalities. Furan2-ylmethylene-Hydrazide (FATH) achieves one of the highest inhibition efficiencies (99.9 %) for carbon steel in a weaker acidic environment (0.1 M HCl) at 0.6 mM, showing remarkable performance but at a lower acid concentration. 4-(2-Mercapto-1,3,4-oxadiazole-5-yl)pyridine (MOP), similar to NFS, offers high inhibition efficiency (93.6 %) for mild steel in 1.0 M HCl at the same concentration (0.5 mM), highlighting its effectiveness with sulfur-containing groups in its structure. Hydroxymethyl Furfural (HMF) demonstrates good inhibition at 89.0 % efficiency at a lower concentration (0.3 mM), providing an alternative with slightly lower efficiency but at a lower dosage, indicating its high potency.

NFS provides a strong inhibition efficiency (92.1 %) at just 0.5 mM in 1.0 M HCl, which is competitive with other high-performing inhibitors such as MOP. NFS's dual adsorption mechanism (physisorption and chemisorption) enhances surface coverage and stability at low concentrations. NFS remains effective even at elevated temperatures, an

Table 3Comparative Performance of Organic Corrosion Inhibitors with Furan, Hydrazine, and Related Functionalities in Acidic Solutions.

Inhibitor	Acidic solution	IE%	Metal	Conc. (mM)	Reference
NFS	1.0 M HCl	92.1	Mild steel	0.5	This study
Furan-2-ylmethylene- hydrazide (FATH)	0.1 M HCl	99.9	Carbon steel	0.6	[45]
(2E)-2-(Furan-2- ylmethylidene) hydrazine Carbothioamide	0.5 M H ₂ SO ₄	60.0	Carbon steel	1.5	[46]
5-(2- Hydroxybenzylidene)- hydrazine derivative (HBHD)	1.0 M HCl	88.5	Mild steel	1.0	[47]
4-(2-Mercapto-1,3,4- oxadiazole-5-yl) pyridine (MOP)	1.0 M HCl	93.6	Mild steel	0.5	[48]
Furan-2-carbaldehyde (FF1)	1.0 M HCl	87.0	Mild steel	1.0	[49]
Hydroxymethyl furfural (HMF)	1.0 M HCl	89.0	Mild steel	0.3	[50]

essential feature for industrial applications, whereas some inhibitors like HBHD perform optimally only at specific temperatures. The combination of physisorption and chemisorption for NFS enables it to form a stable protective layer on the mild steel surface, comparable to FATH and MOP. Despite its high efficiency, NFS contains halogen atoms, raising potential concerns about toxicity and environmental impact. Green inhibitors, although slightly less efficient, such as Henna extract or other natural alternatives, offer a more environmentally friendly option. NFS proves to be an efficient inhibitor in acidic environments, particularly for mild steel in hydrochloric acid solutions, and stands out due to its high performance at low concentrations. While similar inhibitors like FATH and MOP also show excellent efficiency, further studies are recommended to assess NFS's potential for broader applications, its environmental impact, and its performance in various corrosive media.

3.12. Suggested inhibition mechanism of NFS for mild steel in acidic solutions

The corrosion inhibition of mild steel in acidic environments by NFS is primarily attributed to the adsorption of NFS molecules onto the steel surface, forming a protective barrier that impedes corrosive reactions. The inhibition mechanism includes the following key elements [41–43]:

- Adsorption and Film Formation: NFS molecules adsorb onto the steel surface through both physisorption (electrostatic interactions) and chemisorption (coordinate covalent bonding), facilitated by nitrogen and bromine atoms in its structure. This adsorption creates a compact protective layer that blocks the metal surface from aggressive ions such as chloride (Cl⁻), thereby reducing the corrosion rate.
- Blocking of Active Sites: Adsorbed NFS molecules cover active corrosion sites on the metal, limiting the anodic (metal dissolution) and cathodic (hydrogen evolution) reactions. This dual inhibition effect classifies NFS as a mixed-type inhibitor.
- 3. Thermodynamic Insights: The adsorption of NFS follows the Langmuir isotherm, with Gibbs free energy values suggesting both physisorption and chemisorption. Increasing concentrations of NFS lead to higher inhibition efficiency by forming a denser and more continuous protective film, as evidenced by increased R_{ct} and decreased double-layer capacitance.
- Competitive Adsorption: In acidic media, NFS molecules displace chloride ions from the metal surface, further enhancing protection by reducing aggressive ion access to the metal.

This combined mechanism of physisorption and chemisorption by NFS results in a stable and effective corrosion inhibition layer on mild steel in acidic conditions. In addition to the adsorption of NFS onto the mild steel surface, there is a possibility of complex formation between NFS and Fe(II) ions. This occurs because the nitrogen and oxygen atoms in NFS possess lone pairs of electrons that can coordinate with Fe(II) ions, potentially forming stable NFS-Fe(II) complexes. Such complex formation can enhance the protective barrier on the metal surface, as the NFS-Fe(II) complexes further reduce the availability of active sites for corrosive reactions. The presence of these complexes likely strengthens the chemisorption interactions between NFS and the steel surface, contributing to the overall stability and effectiveness of the corrosion inhibition layer. Fig. 7 demonstrates how NFS molecules adsorb onto the mild steel surface, forming a protective layer that blocks aggressive chloride ions (Cl⁻) and reduces both anodic and cathodic reactions, thus inhibiting corrosion.

4. Conclusion

The study on NFS as a corrosion inhibitor for mild steel in acidic environments successfully demonstrates its efficacy using Electrochemical Impedance Spectroscopy, weight loss measurements,

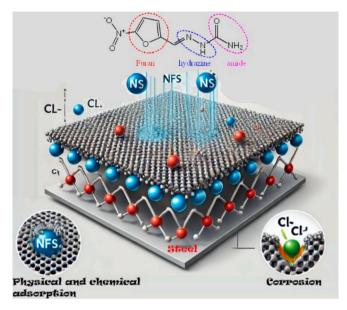


Fig. 7. Suggested Mechanism of Corrosion Inhibition by 5-Nitro-2-furaldehyde Semicarbazone (NFS) on Mild Steel in Acidic Environments.

adsorption isotherms, and Scanning Electron Microscopy. The findings reveal that NFS shows a high inhibition efficiency, reaching up to 92.1 % at 0.5 mM concentration based on weight loss measurements. NFS forms a protective layer on the mild steel surface, significantly reducing the corrosion rate and improving protection with increasing concentrations. The adsorption behavior aligns with the Langmuir isotherm, and both physisorption and chemisorption mechanisms contribute to its effectiveness. Additionally, NFS exhibits better performance at higher temperatures, highlighting its potential for industrial applications in environments with fluctuating thermal conditions. Despite its efficiency, further research into its environmental impact and performance in nonacidic media could enhance its industrial relevance. This study adds valuable insights into the application of NFS as a cost-effective and environmentally friendly corrosion inhibitor in corrosive solutions environments, especially in industrial applications where temperature variations are common. However, further investigation into its environmental impact and performance in various acidic environments is recommended to improve its efficient usage across different industries.

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CRediT authorship contribution statement

Dalia M. Jamil: Visualization, Validation, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **H.S. Aljibori:** Conceptualization, Project administration, Resources, Validation, Writing – original draft. **Ahmed Alamiery:** Writing – review & editing, Writing – original draft, Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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