

Adsorption, kinetic, thermodynamic, thermal stability, and RSM optimization studies of *Mitragyna speciosa* as a green corrosion inhibitor for API 5 L X65 steel in aggressive oil and gas well treatment fluids

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ABSTRACT

The present study reports, for the first time, the use of *Mitragyna speciosa* leaf extract (MSLE) as a sustainable corrosion inhibitor for API 5 L X65 pipeline steel under high-temperature acidic conditions representative of oil and gas well treatment environments. An integrated approach combining adsorption modeling, kinetic–thermodynamic evaluation, thermal stability assessment (TGA/DTA), and response surface methodology (RSM) optimization was employed to provide comprehensive mechanistic insight into inhibitor performance at elevated temperatures (323–343 K), concentrations of 0.2–0.8 g/L, and immersion times of 2–10 h. Adsorption behavior was evaluated using Langmuir, Freundlich, and Temkin isotherms ($R^2 = 0.942\text{--}0.995$), demonstrating strong model agreement and confirming spontaneous adsorption through negative ΔG_{ads} values. The thermodynamic parameters indicate a mixed adsorption mechanism involving both physical and chemical interactions. The presence of MSLE increased the apparent activation energy relative to the uninhibited system, confirming adsorption-mediated surface blocking. Transition state analysis revealed an endothermic activation process accompanied by reduced interfacial disorder in the inhibited system. Thermogravimetric analysis showed negligible mass loss below 120 °C, corresponding to moisture and volatile removal, while major decomposition occurred between 250 and 380 °C, confirming adequate thermal stability under corrosion-testing and oilfield acidizing conditions. RSM analysis demonstrated satisfactory predictive capability ($R^2 = 0.8098$), with inhibitor concentration identified as the dominant factor influencing inhibition efficiency. The maximum experimental inhibition efficiency of 98.40% was achieved at 0.8 g/L, 333 K, and 2 h, while numerical optimization predicted optimal operating conditions at 0.8 g/L, 343 K, and 9.13 h with a validated efficiency of 90.24%. The novelty of this work lies in the integrated thermodynamic–kinetic–thermal–statistical evaluation of MSLE under elevated temperature oilfield conditions. This comprehensive framework not only elucidates the inhibition mechanism but also establishes a systematic basis for optimizing the performance of plant-derived corrosion inhibitors in aggressive acidic environments, contributing to the advancement of sustainable corrosion mitigation strategies.

1. Introduction

Carbon steels, particularly high-strength pipeline grades such as API 5 L X65 steel, are widely used in the oil and gas industry due to their excellent mechanical strength, weldability, cost-effectiveness, and ability to withstand severe service environments [1–6]. During well treatment operations, including acidizing, stimulation, descaling, and pickling, these steels are exposed to highly aggressive acidic solutions. Hydrochloric acid-based fluids are commonly employed to enhance reservoir permeability and remove mineral deposits; however, their

strong corrosive nature significantly accelerates metal dissolution, leading to structural degradation, operational failure, environmental risks, and substantial economic losses [7–12]. Consequently, controlling acid-induced corrosion in such environments remains a critical challenge in oilfield operations.

Among the available mitigation strategies, the use of corrosion inhibitors is widely recognized as one of the most practical and cost-effective approaches for reducing steel degradation in acidic media [13–17]. Organic inhibitors typically function through adsorption onto the metal surface, forming a protective film that suppresses anodic metal

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dissolution and/or cathodic hydrogen evolution reactions [18,19]. The effectiveness of these inhibitors depends strongly on their adsorption characteristics, thermodynamic feasibility, kinetic behavior, and stability under operational conditions [20,21]. Therefore, comprehensive evaluation of adsorption isotherms, activation parameters, and thermodynamic functions is essential to understand inhibitor–metal interactions and to predict performance in industrial systems. Conventional inhibitors used in acidic oilfield environments are often synthetic organic compounds containing heteroatoms such as nitrogen, sulfur, oxygen, and phosphorus, along with conjugated π -electron systems [22]. Although these compounds demonstrate high inhibition efficiency, many pose environmental and regulatory concerns due to toxicity, limited biodegradability, and ecological persistence [23]. As a result, increased environmental awareness and stricter regulations have intensified the search for sustainable and environmentally benign alternatives derived from renewable resources.

Recent advancements in green corrosion inhibition have extended beyond traditional plant extracts to include nanostructured carbon-based materials. Zhao et al. [24]. investigated phosphorus-doped carbon quantum dots as inhibitors in neutral Mg–air battery systems and demonstrated that heteroatom doping enhances adsorption strength and suppresses anodic corrosion through modulation of interfacial electron transfer processes. Similarly, Wan et al. [25]. studied N,S-co-doped carbon quantum dots as environmentally friendly inhibitors for brine heaters in multistage flash desalination systems, attributing their performance to synergistic heteroatom coordination and stable surface film formation. Tan et al. [26]. further provided mechanistic insight into microwave-assisted synthesized carbon quantum dots as inhibitors for copper corrosion in sulfuric acid, highlighting the roles of surface functional groups, charge transfer interactions, and adsorption-controlled electrochemical suppression. These studies underscore the importance of heteroatom-containing functional groups and adsorption-mediated mechanisms in advanced sustainable corrosion inhibitor systems.

Despite the promising performance of doped carbon quantum dots, their synthesis often requires controlled laboratory conditions, energy-intensive preparation routes, or specialized precursors, which may limit large-scale industrial implementation, particularly in oilfield applications. In contrast, plant-derived extracts represent abundant, low-cost, and biodegradable alternatives rich in naturally occurring heteroatom-containing compounds capable of surface adsorption [27–29]. Plant extracts are typically rich in alkaloids, flavonoids, tannins, polyphenols, and saponins containing hydroxyl, carbonyl, amine, and aromatic functional groups that facilitate adsorption through electrostatic attraction, donor–acceptor interactions, and coordination bonding with metal surfaces. Numerous studies have demonstrated that such extracts exhibit thermodynamically favorable adsorption and kinetically efficient inhibition behavior in acidic environments [30,31]. However, many investigations remain limited to evaluating inhibition efficiency under near-ambient temperature conditions, without incorporating detailed thermodynamic analysis, thermal stability assessment, and statistical optimization under elevated temperatures relevant to oilfield operations.

Mitragyna speciosa, a medicinal plant widely distributed in tropical regions, is known for its high alkaloid and polyphenolic content. Although extensively studied from a pharmacological perspective, its corrosion inhibition potential particularly under aggressive oil and gas well treatment conditions remains largely unexplored [32]. The chemical complexity of *Mitragyna speciosa* leaf extract suggests strong adsorption capability and potential thermal stability, making it a promising candidate for high-temperature acidic systems. A mechanistic understanding of corrosion inhibition requires more than simple measurement of inhibition efficiency. Determination of activation energy, enthalpy and entropy of activation, and Gibbs free energy of adsorption provides insight into the nature of the adsorption process whether physical, chemical, or mixed its temperature dependence, and its

stability. Furthermore, thermogravimetric and differential thermal analyses are necessary to evaluate inhibitor stability under elevated temperatures typical of acidizing operations. In addition, optimization of corrosion systems requires systematic investigation of interacting operational parameters. Response Surface Methodology has emerged as a powerful statistical tool for modeling nonlinear relationships between process variables and inhibition efficiency, enabling efficient experimental design and prediction of optimal operating conditions with reduced experimental effort [33].

In the present study, *Mitragyna speciosa* leaf extract is investigated as a green corrosion inhibitor for API 5 L X65 steel in 1.0 M HCl under elevated temperature conditions representative of oilfield acidizing environments. The work integrates adsorption isotherm modeling, kinetic and thermodynamic evaluation using Arrhenius and transition state analyses, thermal stability assessment through TGA/DTA, and statistical optimization using Box–Behnken response surface methodology. By positioning plant-derived phytochemical systems within the broader context of advanced green inhibitors such as heteroatom-doped carbon quantum dots [24–26], this study provides a comprehensive thermodynamic, kinetic, thermal, and statistical framework for evaluating sustainable corrosion mitigation strategies in high-temperature acidic environments.

2. Materials and methods

2.1. Materials

API 5 L X65 pipeline steel was used as the substrate material in this study. Its chemical composition (wt. %) is as follows: 0.081 % C, 0.287 % Si, 1.051 % Mn, 0.026 % P, 0.012 % S, 0.038 % Al, 0.019 % Cr, 0.056 % V, 0.044 % Nb, with the balance being Fe.

2.2. Preparation of *Mitragyna speciosa* extract

Fresh leaves of *Mitragyna speciosa* were collected from Kurutie community, Gbaramatu, Warri South-West Local Government Area, Delta State, Nigeria. The plant material was authenticated prior to use. The collected leaves were thoroughly washed with distilled water to remove adhering dust, soil particles, and other contaminants, and then air-dried under ambient laboratory conditions for 48 h. Subsequently, the leaves were oven-dried at 50 °C for 72 h to eliminate residual moisture and prevent thermal degradation of thermolabile phytochemical constituents. The dried leaves were pulverized using a laboratory mechanical grinder and passed through a 100-mesh sieve to obtain a uniform fine powder, thereby increasing surface area and enhancing extraction efficiency. Extraction was performed using the maceration technique with methanol as the solvent due to its effectiveness in extracting alkaloids, polyphenols, and other bioactive compounds.

A measured quantity of 120 g of the powdered leaf material was immersed in 1000 mL of analytical-grade methanol, maintaining a solid-to-solvent ratio of 1:10 (w/v). The mixture was magnetically stirred for 2 h to ensure homogeneity and facilitate solvent penetration into the plant matrix. The suspension was then allowed to macerate for 72 h at room temperature, with intermittent stirring every 6 h to maximize phytochemical extraction through diffusion and solvent–solute interaction. After maceration, the mixture was filtered using Whatman No 1 filter paper to separate the solid residue from the extract solution. The filtrate was concentrated using a rotary evaporator under reduced pressure at 45 °C to remove excess solvent and reduce the volume to approximately 30 % of the original volume. The resulting semi-solid concentrate was further oven-dried at 50 °C for 72 h to remove residual solvent and obtain a dry crude extract. The crude *Mitragyna speciosa* leaf extract (MSLE) was stored in airtight glass containers and kept in a desiccator to prevent moisture absorption prior to use. Stock solutions were prepared by dissolving appropriate quantities of MSLE in 1.0 M HCl to obtain inhibitor concentrations in the range of 0.2–0.8 g/L. All

solutions were freshly prepared before each experiment to ensure consistency and reproducibility. To confirm the presence of functional groups responsible for corrosion inhibition, Fourier Transform Infrared (FTIR) analysis was performed on the crude extract prior to corrosion testing.

2.3. Aggressive solution preparation

A 1.0 M hydrochloric acid (HCl) solution was prepared by diluting analytical-grade 37 % concentrated HCl with distilled water in a 1000 mL volumetric flask.

2.4. FTIR study

Fourier Transform Infrared (FTIR) spectroscopy was employed to analyze the crude MSLE and the Fe-MSLE complex obtained from the desorbed protective film on X65 steel. The analyses were carried out using a Nicolet iS5 FT-IR spectrometer (Thermo Fisher Scientific, USA) with the KBr pellet technique. The spectra were recorded over the wavenumber range of 400–4000 cm^{-1} to identify functional groups responsible for adsorption and corrosion inhibition mechanisms [34].

2.5. Thermogravimetric and differential thermal analysis (TGA/DTA)

Thermogravimetric and differential thermal analyses (TGA/DTA) of the MSLE were performed using a DTG-60H instrument (Shimadzu, Japan). Approximately 10 mg of the dried extract was heated from room temperature to 1000 °C at a heating rate of 10 °C/min under a nitrogen atmosphere.

2.6. Weight loss method

MSLE was evaluated for its corrosion inhibition performance on X65 pipeline steel in 1.0 M hydrochloric acid solution using the weight loss method. Prior to immersion, X65 steel coupons were mechanically polished with successive grades of silicon carbide abrasive papers, degreased with acetone, thoroughly rinsed with distilled water, and air-dried. Pre-weighed steel specimens were fully immersed in 250 mL of the test solution using a non-reactive suspension thread. The experiments were conducted in both the absence and presence of varying concentrations of MSLE. The beakers containing the test solutions were placed in a thermostatically controlled water bath and maintained at 323, 333, and 343 K for immersion periods of 2, 4, 6, 8, and 10 h. At the end of each immersion period, the specimens were removed from the solution, carefully rinsed with distilled water to remove loosely adhered corrosion products, washed with methanol, and air-dried. The cleaned specimens were reweighed using an analytical balance. For each temperature investigated, the experiments were performed in triplicate to ensure accuracy and reproducibility. The weight loss (Δw) was determined as the difference between the initial and final weights of the specimens. The corrosion rate (CR), inhibition efficiency (IE %), and surface coverage (θ) were subsequently calculated from the measured weight loss values using Eqs. (1–4), as reported in the literature [35–37].

$$\Delta w = W_i - W_f \quad (1)$$

Where W_i and W_f are the initial and final weights of the coupon, respectively.

The corrosion rate (CR) was calculated using Eq. (2):

$$CR = \frac{\Delta w}{A \times t} \quad (2)$$

where Δw is the weight loss (g), A is the surface area of the coupon (cm^2), and t is the immersion time (h).

The inhibition efficiency (IE %) was determined using Eq. (3):

$$IE(\%) = \frac{CR_0 - CR_i}{CR_0} \times \frac{100}{1} \quad (3)$$

where CR_0 and CR_i are the corrosion rates in the absence and presence of MSLE, respectively.

The surface coverage (θ) was calculated using Eq. (4):

$$\theta = \frac{CR_0 - CR_i}{CR_0} \quad (4)$$

2.7. Adsorption parameters and thermodynamic studies

Adsorption plays a crucial role in elucidating the corrosion inhibition mechanism of organic inhibitors. Corrosion inhibitors mitigate metal dissolution by adsorbing onto active sites on the metal surface through physical (physisorption), chemical (chemisorption), or mixed adsorption mechanisms. This adsorption process leads to the formation of a protective barrier that suppresses anodic metal dissolution and the cathodic hydrogen evolution reaction. To better understand the adsorption behavior of MSLE on the surface of X65 steel in 1.0 M HCl solution, the surface coverage (θ) was calculated from weight loss measurements. The calculated surface coverage values were subsequently used to evaluate different adsorption isotherm models. The experimental data were fitted to the Langmuir, Temkin, and Freundlich adsorption isotherms in order to determine the most appropriate adsorption model and to gain insight into the nature of the inhibitor–metal surface interactions [38].

The Langmuir adsorption isotherm assumes monolayer adsorption on a homogeneous surface with no interaction between adsorbed species and is expressed as:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad (5)$$

where C is the inhibitor concentration (g L^{-1}), θ is the surface coverage, and K_{ads} is the equilibrium adsorption constant.

The Temkin adsorption isotherm considers interactions between adsorbed molecules and assumes that the heat of adsorption decreases linearly with surface coverage. It is given by:

$$\theta = \frac{1}{a} \ln[K_{ads} C] \quad (6)$$

where a is the interaction parameter representing lateral interactions between adsorbed species, and K_{ads} is the Temkin adsorption constant.

The Freundlich adsorption isotherm describes adsorption on heterogeneous surfaces and is expressed as:

$$\ln \theta = \ln K_{ads} + \frac{1}{n} \ln C \quad (7)$$

where n is the heterogeneity factor indicating adsorption intensity, and K_{ads} is the Freundlich adsorption constant.

The best fitting adsorption isotherm was chosen from the plots which contain the linear regression coefficient (R^2). The thermodynamic parameters of adsorption were also assessed using the adsorption equilibrium constant. The standard free energy of adsorption (ΔG_{ads}) was determined by the following expression:

$$\Delta G_{ads} = -RT \ln[55.5 K_{ads}] \quad (8)$$

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

The standard enthalpy (ΔH_{ads}) and entropy (ΔS_{ads}) of adsorption were determined using the Van't Hoff relation:

$$\ln K_{ads} = \frac{\Delta H_{ads}}{RT} + \frac{\Delta S_{ads}}{R} \quad (9)$$

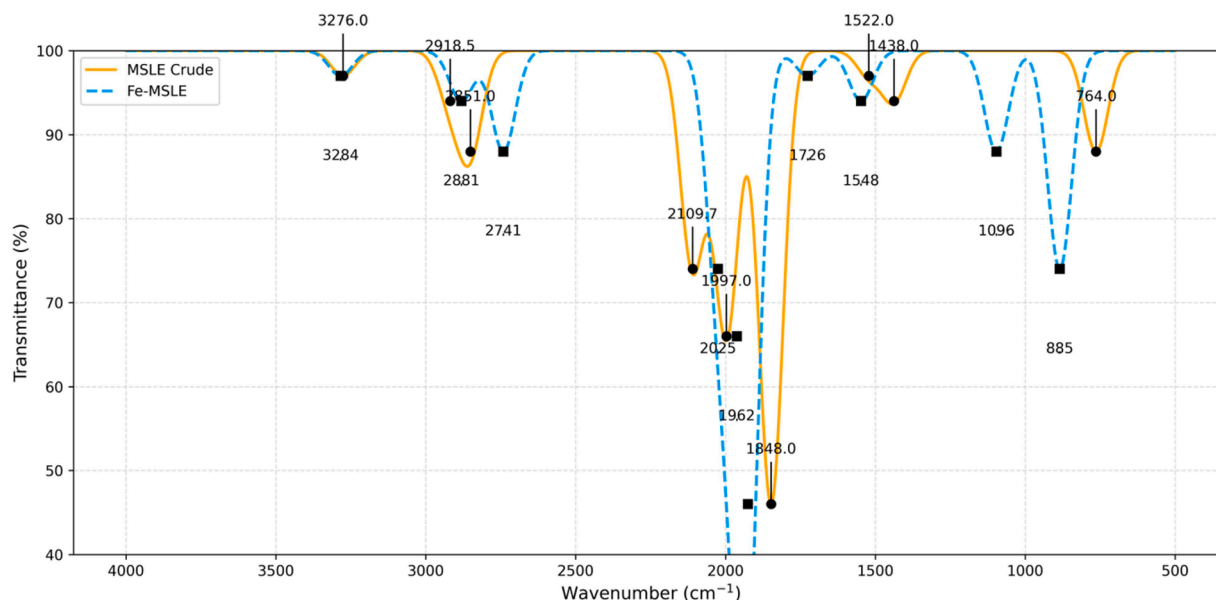


Fig. 1. FTIR spectrum of MSLE and Fe-MSLE.

A plot of $\ln K_{ads}$ versus $1/T$ yields a straight line, from which ΔH_{ads} and ΔS_{ads} were obtained from the slope and intercept, respectively.

Negative values of ΔG_{ads} indicate a spontaneous adsorption process. Values around -20 kJ mol^{-1} generally suggest physisorption, while values more negative than -40 kJ mol^{-1} are typically associated with chemisorption. The sign and magnitude of ΔH_{ads} provide insight into the thermal nature of the adsorption process, distinguishing between exothermic and endothermic behavior. Meanwhile, ΔS_{ads} reflects changes in the degree of disorder at the metal–solution interface during adsorption.

The effect of temperature on the corrosion rate was analyzed using the Arrhenius equation:

$$\ln (CR) = \ln A - \frac{E_a}{RT} \quad (10)$$

where CR is the corrosion rate, A is the pre-exponential factor, E_a is the apparent activation energy of the corrosion process, R is the universal gas constant, and T is the absolute temperature.

A plot of $\ln(CR)$ versus $1/T$ yields a straight line, from which the apparent activation energy (E_a) was calculated using the slope ($-E_a/R$). An increase in E_a in the presence of MSLE compared to the uninhibited solution indicates the formation of an energy barrier due to inhibitor adsorption, which is commonly associated with a surface-blocking mechanism and is often characteristic of physical adsorption. Further insight into the corrosion inhibition mechanism was obtained using transition state theory. The transition state equation is expressed as:

$$CR = \frac{RT}{Nh} + \exp\left(\frac{\Delta S}{R}\right) \exp\left(-\frac{\Delta H}{RT}\right) \quad (11)$$

where ΔH and ΔS are the enthalpy and entropy of activation, respectively, N is Avogadro's number, and h is Planck's constant. Rearranging Eq. (12) gives:

$$\ln \frac{CR}{T} = \ln \left(\frac{R}{Nh} \right) + \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (12)$$

A plot of $\ln(CR/T)$ versus $1/T$ produces a straight line, from which the enthalpy of activation (ΔH) and entropy of activation (ΔS) were determined from the slope ($-\Delta H/R$) and intercept, respectively. In this study, positive values of ΔH indicate that the corrosion process is endothermic in nature. Conversely, negative values of ΔS suggest a

decrease in disorder during the formation of the activated complex, which is consistent with an associative adsorption mechanism involving inhibitor molecules at the metal–solution interface.

2.8. Response surface methodology (RSM) study

Response modeling and experimental design were carried out using Design-Expert version 13 software. The process variables investigated included immersion time, temperature, and inhibitor concentration for X65 steel in 1.0 M HCl solution. Inhibition efficiency (IE, %) was selected as the response variable. A Box–Behnken design (BBD) was employed, and a total of 60 experimental runs were generated and conducted in randomized order to minimize systematic bias. The relationship between the independent variables and inhibition efficiency was established by developing an empirical regression model, in accordance with established statistical methodologies reported in the literature [31,36].

3. Results and discussion

3.1. FTIR analysis

The FTIR spectra of the crude MSLE extract and the Fe–MSLE complex, as presented in Fig. 1, exhibit several characteristic absorption bands that confirm the presence of multiple functional groups responsible for the chemical nature of the extract and its interaction with iron species. A comparative analysis of both spectra reveals noticeable peak shifts, variations in intensity, and the appearance or disappearance of certain bands, indicating coordination or interaction between the extract constituents and iron. A broad absorption band observed in the region 3276–3284 cm^{-1} in both spectra is attributed to O–H stretching vibrations, typically associated with alcohols and phenolic compounds. The slight shift from 3284 cm^{-1} in the crude MSLE to 3276 cm^{-1} in the Fe–MSLE spectrum suggests the involvement of hydroxyl groups in coordination with Fe^{2+} ions, resulting in modifications in hydrogen bonding strength. The bands at 2918–2851 cm^{-1} in the crude extract and 2881–2741 cm^{-1} in the Fe–MSLE spectrum correspond to C–H stretching vibrations of aliphatic $-\text{CH}_2$ and $-\text{CH}_3$ groups. The observed reduction in intensity and slight displacement of these peaks after interaction with iron indicate possible involvement of aliphatic chains in surface interaction and structural rearrangement [33].

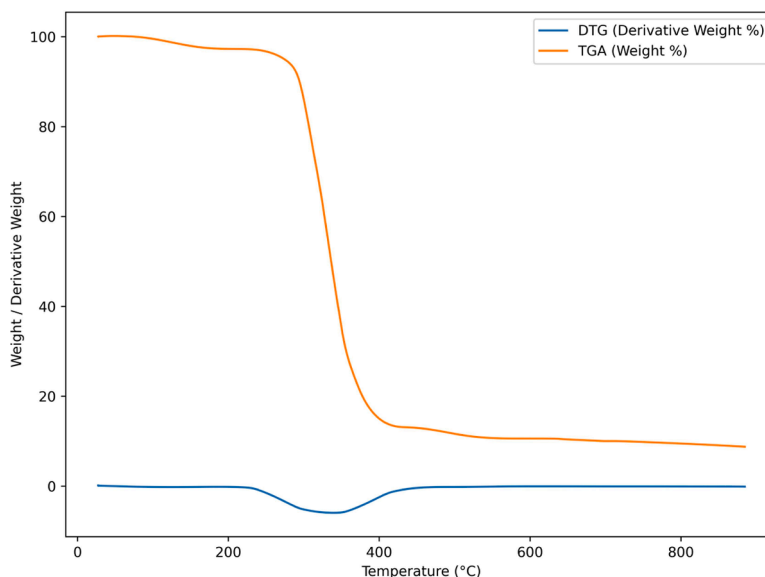


Fig. 2. TGA/DTG plots of MSLE.

A strong absorption band in the range $2109\text{--}1997\text{ cm}^{-1}$ for the crude MSLE and $2025\text{--}1925\text{ cm}^{-1}$ for Fe-MSLE may be assigned to $\text{C}\equiv\text{C}$ or $\text{C}\equiv\text{N}$ stretching vibrations or combination bands. The noticeable shift and intensity changes in the Fe-MSLE spectrum further support the participation of these functional groups in metal-ligand interactions. Additionally, a prominent band centered at 1848 cm^{-1} in the crude extract, which shifts and intensifies in the Fe-MSLE spectrum (approximately $1962\text{--}1726\text{ cm}^{-1}$), is attributed to $\text{C}=\text{O}$ stretching vibrations of carbonyl-containing compounds such as ketones, aldehydes, or esters. The shift toward lower wavenumbers in the Fe-MSLE spectrum suggests coordination between carbonyl oxygen atoms and iron ions, indicating a reduction in bond strength due to complex formation. The bands at 1522 and 1438 cm^{-1} in the crude MSLE and 1548 and 1726 cm^{-1} in the Fe-MSLE spectrum correspond to $\text{C}=\text{C}$ stretching vibrations of aromatic rings and O-H bending modes. Shifts in peak positions and intensity changes after interaction with iron indicate possible π -electron interactions and chelation involving aromatic and phenolic groups. Furthermore, the appearance of bands at 1096 and 885 cm^{-1} in the Fe-MSLE spectrum, compared to the band at 764 cm^{-1} in the crude

extract, can be associated with C-O stretching vibrations and Fe-O bond formation. The emergence and strengthening of these low-wavenumber bands provide strong evidence for metal-oxygen bond formation. Overall, the FTIR analysis confirms that MSLE contains multiple functional groups, including hydroxyl, carbonyl, aliphatic, and aromatic moieties, which actively participate in coordination with iron. The observed peak shifts, intensity variations, and formation of new bands in the Fe-MSLE spectrum confirm metal-ligand interactions and support the proposed adsorption mechanism. These findings validate the potential of MSLE as an effective corrosion inhibitor and surface-modifying agent [37].

3.2. Thermal stability of MSLE

The TGA-DTG curves of MSLE, as shown in Fig. 2, reveal distinct thermal decomposition stages that explain its suitability as a temperature-resistant corrosion inhibitor for X65 steel in 1.0 M HCl. In the initial stage, from approximately 30 to $120\text{ }^{\circ}\text{C}$, only minimal weight loss is observed in the TGA curve. This region corresponds to the removal of physically adsorbed moisture and low-boiling volatile

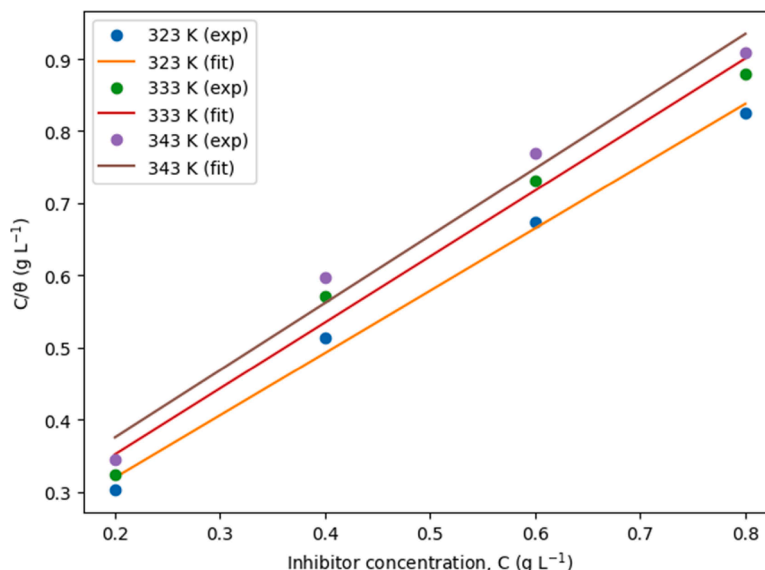


Fig. 3. Langmuir adsorption isotherm of X65 steel in 1 M HCl at vary temperatures.

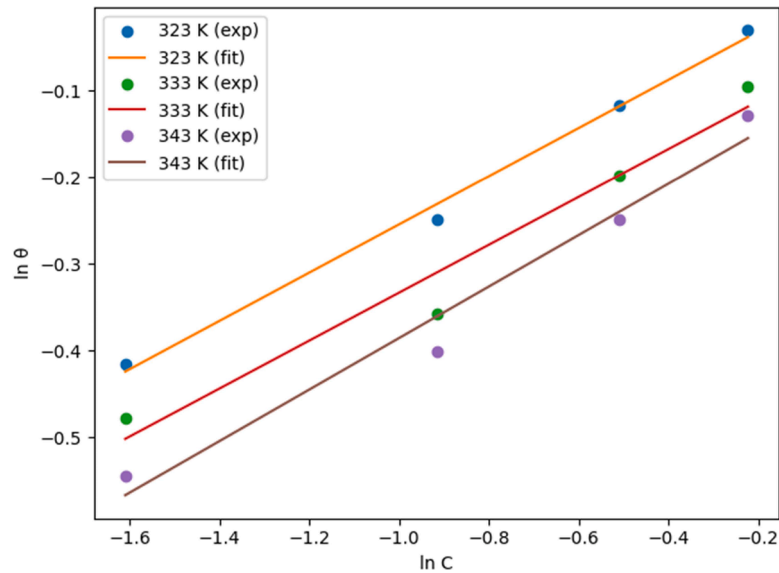


Fig. 4. Freundlich adsorption isotherm of X65 steel in 1 M HCl at vary temperatures.

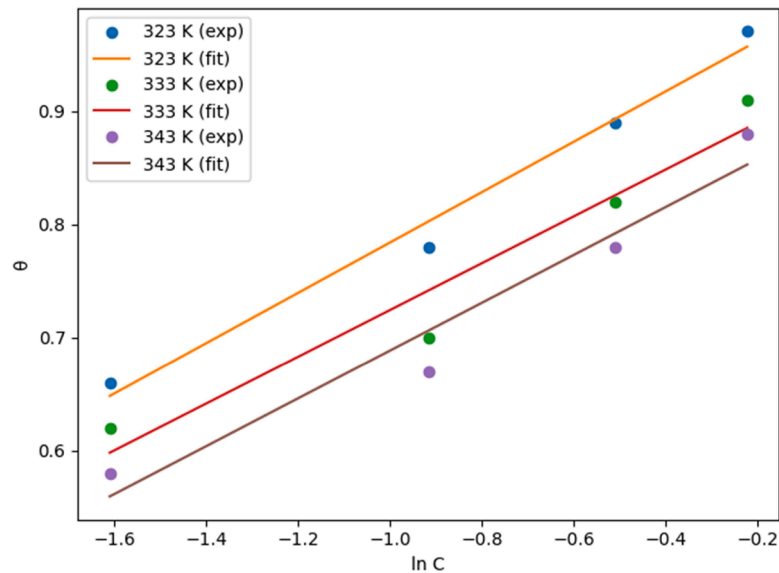


Fig. 5. Temkin adsorption isotherm of X65 steel in 1 M HCl at vary temperatures.

components, indicating that MSLE remains thermally stable within the temperature range commonly used for corrosion testing and acid pickling processes. As the temperature increases beyond this range, the extract maintains relatively high thermal stability up to about 250 °C.

The major thermal degradation occurs between approximately 250 and 380 °C, where a pronounced weight loss is observed in the TGA curve, accompanied by a distinct DTG peak centered around 320–350 °C. This primary degradation stage is attributed to the decomposition of organic

Table 1
Adsorption isotherms parameters for MSLE.

Isotherms	Tempt (K)	R2	Slope	Intercept	Kads	n	a	ΔGads
Langmuir	323	0.994	0.864	0.147	5.878			-4.76
	333	0.983	0.915	0.169	7.827			-5.7
	343	0.982	0.932	0.189	4.93			-4.55
Freundlich	323	0.995	0.278	0.024	1.057	3.597		-0.148
	333	0.962	0.277	-0.057	0.877	3.61		-0.3634
	343	0.971	0.297	-0.088	1.225	3.367		-0.5778
Temkin	323	0.985	0.222	1.006	4.527		4.5	-4.05
	333	0.942	0.207	0.931	4.500		4.83	-4.164
	343	0.945	0.211	0.900	4.266		4.74	-4.133

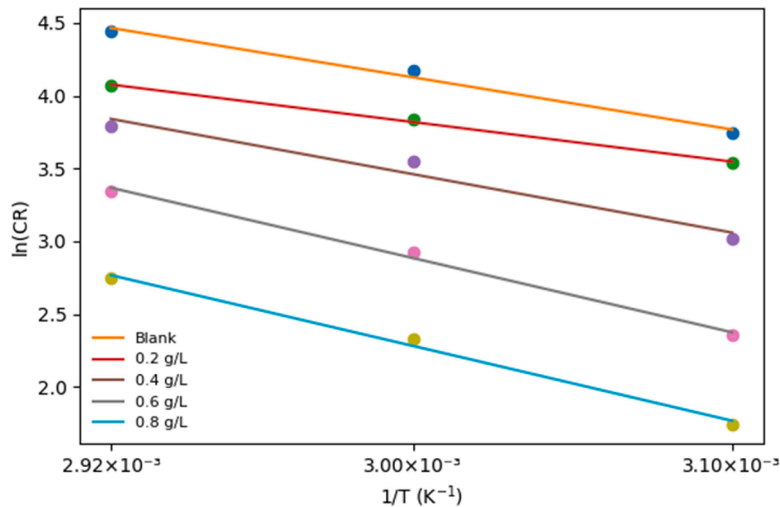


Fig. 6. Arrhenius plot of X65 steel in 1 M HCl the absence and presence of MSLE.

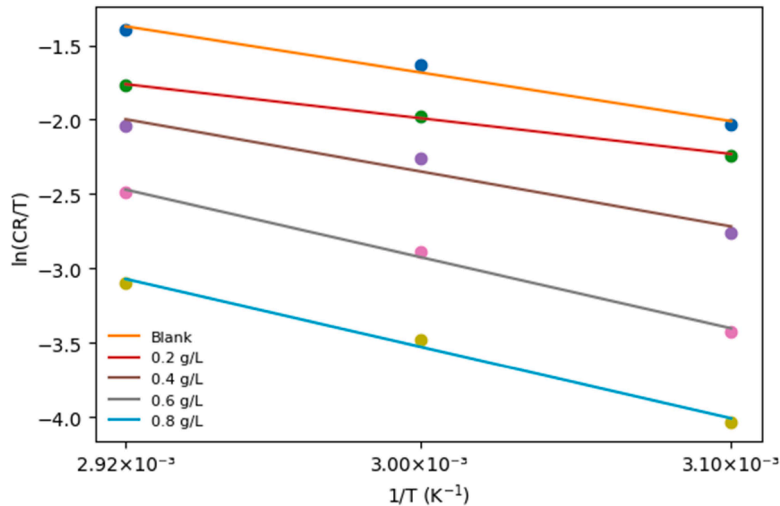


Fig. 7. Transition state plots of X65 steel in 1 M HCl the absence and presence of MSLE.

constituents such as alkaloids, polyphenols, and other heteroatom-containing compounds responsible for corrosion inhibition. The presence of a sharp DTG peak indicates that the active phytochemical components decompose within a well-defined temperature range rather than gradually, suggesting structural integrity and stability at lower and moderate temperatures. These findings confirm that MSLE possesses sufficient thermal resilience for corrosion protection applications under the investigated temperature conditions [33].

3.3. Adsorption isotherm behaviour and thermodynamic feasibility

The adsorption behavior of MSLE on X65 steel was evaluated using the Langmuir, Freundlich, and Temkin isotherm models (Figs. 3–5; Table 1). All three models exhibited high coefficients of determination ($R^2 = 0.942\text{--}0.995$), indicating that the experimental data are well described by these adsorption models according to their respective assumptions. For the Langmuir model (Fig. 3), the adsorption equilibrium constants (K_{ads}) were determined to be 5.878, 7.827, and 4.93 at 323, 333, and 343 K, respectively. The corresponding ΔG_{ads} values were negative (-4.55 to -5.70 kJ mol⁻¹), confirming spontaneous adsorption and strong affinity of MSLE constituents toward the steel surface. The

Table 2
Thermodynamic parameters for MSLE.

S. No	MSLE (System) Conc.(g/L)	Ea (KJ/mol)	-ΔG _{ads} (KJ/mol)			ΔH _{ads} (KJ/mol)	ΔS _{ads} (KJ/mol)
			323K	333K	343K		
1	0	32.01	69.20	70.44	71.67	29.24	-123.71
2	0.2	24.30	69.80	71.29	72.79	21.54	-149.41
3	0.4	35.85	71.10	72.28	73.45	33.08	-117.71
4	0.6	45.57	72.94	73.87	74.81	42.80	-93.31
5	0.8	45.86	74.57	75.54	76.52	43.09	-97.45

good linearity of the Langmuir plots suggests predominantly monolayer adsorption with minimal interaction between adsorbed species. As shown in Fig. 4, the Freundlich model yielded heterogeneity factors (n) in the range of 3.367–3.610 ($n > 1$), indicating favorable adsorption on a heterogeneous surface. The associated ΔG_{ads} values were slightly negative (-0.15 to -0.58 kJ mol⁻¹), further supporting spontaneous adsorption, although with weaker adsorption intensity compared to the Langmuir model. The Temkin isotherm (Fig. 5) produced K_{ads} values within a relatively narrow range (4.266–4.527), with corresponding

Table 3

Design matrix and result for inhibiting API 5 L X65 steel corrosion in 1 M HCl with MSLE.

Std	Run	Factor 1 A:Time h	Factor 2 B:Conc g/L	Factor 3 C:Temp K	Response 1 IE %
13	1	2	0.2	323	67.5
54	2	2	0.4	323	77.6
59	3	2	0.6	323	85.9
44	4	2	0.8	323	97.6
49	5	2	0.2	333	67.45
23	6	2	0.4	333	77.32
22	7	2	0.6	333	88.4
58	8	2	0.8	333	98.4
48	9	2	0.2	343	55.3
27	10	2	0.4	343	63.67
55	11	2	0.6	343	69.59
25	12	2	0.8	343	71.34
2	13	4	0.2	323	61.5
40	14	4	0.4	323	72.9
37	15	4	0.6	323	84.2
18	16	4	0.8	323	94.2
20	17	4	0.2	333	65.1
16	18	4	0.4	333	73.1
7	19	4	0.6	333	88.23
50	20	4	0.8	333	93.34
19	21	4	0.2	343	89.4
14	22	4	0.4	343	55.2
57	23	4	0.6	343	68.56
38	24	4	0.8	343	91.56
29	25	6	0.2	323	66.8
51	26	6	0.4	323	73.5
60	27	6	0.6	323	86.8
35	28	6	0.8	323	98.3
34	29	6	0.2	333	59.69
31	30	6	0.4	333	68.68
11	31	6	0.6	333	78.58
45	32	6	0.8	333	89.63
17	33	6	0.2	343	57.36
46	34	6	0.4	343	66.45
47	35	6	0.6	343	78.12
5	36	6	0.8	343	92.23
30	37	8	0.2	323	63.8
33	38	8	0.4	323	78.5
15	39	8	0.6	323	88.3
53	40	8	0.8	323	97.44
6	41	8	0.2	333	58.88
8	42	8	0.4	333	66.38
43	43	8	0.6	333	73.29
26	44	8	0.8	333	89.5
32	45	8	0.2	343	56.48
1	46	8	0.4	343	67.27
3	47	8	0.6	343	68.58
39	48	8	0.8	343	90.45
42	49	10	0.2	323	66
52	50	10	0.4	323	78
56	51	10	0.6	323	88.3
12	52	10	0.8	323	95.44
21	53	10	0.2	333	59.67
24	54	10	0.4	333	66.23
41	55	10	0.6	333	78.5
10	56	10	0.8	333	88.69
28	57	10	0.2	343	54.6
36	58	10	0.4	343	63.67
4	59	10	0.6	343	74.55
9	60	10	0.8	343	88.68

negative ΔG_{ads} values (-4.05 to -4.16 kJ mol $^{-1}$), suggesting thermodynamically feasible adsorption across the studied temperature range [34]. The consistency among the three models indicates that MSLE adsorption involves both surface heterogeneity and intermolecular interactions.

Further insight into the inhibition mechanism was obtained from Arrhenius and transition state analyses (Figs. 6 and 7; Table 2). The

activation energy (E_a) of the uninhibited solution was 32.01 kJ mol $^{-1}$, whereas in the presence of MSLE, E_a increased progressively with inhibitor concentration, reaching 45.86 kJ mol $^{-1}$ at 0.8 g L $^{-1}$ [38–41]. The increase in E_a with inhibitor concentration indicates that MSLE raises the energy barrier of the corrosion reaction, consistent with adsorption-driven blocking of active sites and retardation of charge transfer processes. The calculated enthalpy of activation (ΔH) values were positive (21.54–43.09 kJ mol $^{-1}$), indicating that the corrosion process is endothermic in nature. Meanwhile, the negative entropy of activation (ΔS) values suggest a decrease in disorder during formation of the activated complex, which may be attributed to the establishment of a more ordered inhibitor layer at the metal–solution interface. These thermodynamic parameters collectively support the formation of an adsorbed protective film that suppresses corrosion reactions in the inhibited system [42–45]

3.4. Experimental design matrix and ANOVA of the RSM model

The experimental design matrix and the corresponding inhibition efficiency (IE) values of MSLE in 1.0 M HCl for API 5 L X65 steel, as functions of immersion time (2–10 h), inhibitor concentration (0.2–0.8 g/L), and temperature (323–343 K), are presented in Table 3. The observed IE values varied widely from 54.6 % to 98.4 %, indicating that the selected process variables significantly influence corrosion inhibition performance. Table 4 summarizes the analysis of variance (ANOVA) results for the quadratic RSM model [46]. The model is statistically significant ($F = 23.65$, $p < 0.0001$), confirming that the regression adequately explains the variation in inhibition efficiency within the investigated design space. Among the main effects, inhibitor concentration (B) emerged as the most influential factor ($F = 179.31$, $p < 0.0001$), demonstrating that MSLE dosage predominantly governs surface coverage and corrosion protection efficiency. Temperature (C) was also statistically significant ($F = 26.06$, $p < 0.0001$), consistent with experimental observations that increasing temperature accelerates corrosion reactions and may influence adsorption stability of the inhibitor film.

In contrast, immersion time (A) was not statistically significant ($p = 0.2802$), suggesting that its independent contribution to inhibition efficiency is relatively small compared to concentration and temperature within the studied range. Furthermore, all interaction terms (AB, AC, BC) and quadratic terms (A^2 , B^2 , C^2) were statistically insignificant at the 95 % confidence level ($p > 0.05$), indicating that inhibition efficiency is primarily governed by the linear effects of inhibitor concentration and temperature under the current experimental conditions. The goodness-of-fit statistics demonstrate acceptable predictive capability of the developed model. The coefficient of determination ($R^2 = 0.8098$) indicates that approximately 81 % of the variability in IE is explained by the model. The adjusted R^2 (0.7755) and predicted R^2 (0.7123) show reasonable agreement, confirming model reliability. The relatively low coefficient of variation (C.V. = 8.10 %) reflects good experimental precision, while the high Adeq Precision value (16.76) indicates a satisfactory signal-to-noise ratio and confirms the suitability of the model for navigating the design space [31,36].

3.5. RSM mathematical modeling

The coded regression model developed using Response Surface Methodology (RSM) is presented in Eq. (13). This model enables prediction of the response at any selected combination of independent variables within the investigated experimental domain. The corresponding regression equation expressed in terms of the actual (uncoded) process variables is provided in Eq. (14), allowing direct prediction of the response using real experimental units.

Table 4

RSM analysis of variance (ANOVA) of MSLE IE for curbing API 5 L X65 steel corrosion in 1 M HCl.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	8126.86	9	902.98	23.65	< 0.0001	significant
A-Time	45.51	1	45.51	1.19	0.2802	
B-Conc	6847.26	1	6847.26	179.31	< 0.0001	
C-Tempt	995.21	1	995.21	26.06	< 0.0001	
AB	61.84	1	61.84	1.62	0.2091	
AC	0.7488	1	0.7488	0.0196	0.8892	
BC	70.38	1	70.38	1.84	0.1807	
A ²	3.33	1	3.33	0.0871	0.7691	
B ²	101.30	1	101.30	2.65	0.1097	
C ²	1.30	1	1.30	0.0340	0.8545	
Residual	1909.37	50	38.19			
Cor Total	10,036.23	59				
Std. Dev.	6.18	R²	0.8098			
Mean	76.24	Adjusted R²	0.7755			
C.V. %	8.10	Predicted R²	0.7123			
		Adeq Precision	16.7582			

$$\begin{aligned} \text{IE}\% = & 75.1103 - 1.23167 * A + 14.3324 * B - 4.988 * C \\ & + 1.9262 * AB + 0.1935 * AC - 1.7796 * BC - 0.562857 * A^2 + 2.9235 \\ & * B^2 - 0.312 * C^2 \end{aligned} \quad (13)$$

reported in related studies. The validity of the optimization model was experimentally verified, with the measured inhibition efficiency differing from the predicted value by only 0.78 %. This close agreement confirms the reliability and predictive accuracy of the developed RSM model [36].

$$\begin{aligned} \text{IE} = & -204.235 + -2.29924 * \text{Time} + 203.196 * \text{Inhibitor concentration} \\ & + 1.8467 * \text{Temperature} + 1.60517 * \text{Time} * \text{Inhibitor concentration} \\ & + 0.0048375 * \text{Time} * \text{Temperature} - 0.5932 * \text{Inhibitor concentration} * \text{Temperature} \\ & - 0.0351786 * \text{Time}^2 + 32.4833 * \text{Inhibitor concentration}^2 + -0.00312 * \text{Temperature}^2 \end{aligned} \quad (14)$$

Fig. 8(a–d) presents the response plots for the inhibition efficiency (IE) of MSLE on X65 steel in 1.0 M HCl. As shown in Fig. 8a, the predicted IE values are in good agreement with the experimental results, as evidenced by the near-linear distribution of data points along the parity line. This confirms the adequacy and reliability of the developed model for estimating inhibition efficiency within the investigated design space. The three-dimensional response surface plots (Fig. 8b–d) illustrate the combined effects of inhibitor concentration, temperature, and immersion time on IE. These plots demonstrate that inhibition efficiency is influenced by the interaction of these process variables, with maximum inhibition occurring within a defined optimal region [33]. The response surfaces further confirm that inhibitor concentration exerts the strongest influence, while temperature and immersion time contribute to variations in adsorption stability and corrosion kinetics. Under the optimized conditions, a maximum experimental inhibition efficiency of 98.40 % was achieved, demonstrating the strong protective capability of MSLE and confirming its effectiveness in mitigating the corrosion of X65 steel in 1.0 M HCl solution.

3.6. Process optimization and model validation

Table 5 presents the optimized operating conditions namely inhibitor concentration, immersion time, and temperature for minimizing the corrosion of X65 steel in 1.0 M HCl using MSLE, along with the corresponding inhibition efficiency (IE). Under the optimal conditions, MSLE achieved a maximum inhibition efficiency of 90.24 %, demonstrating its strong protective performance in the acidic medium and confirming its effectiveness as a corrosion inhibitor for X65 steel. The high inhibition efficiency is attributed to the presence of adsorption-active heteroatoms within the phytochemical constituents of MSLE, which facilitate the formation of a stable protective film on the steel surface, as widely

4. Conclusion

This study comprehensively evaluated the corrosion inhibition performance of *Mitragyna speciosa* leaf extract (MSLE) for API 5 L X65 steel in 1.0 M HCl under conditions representative of aggressive oil and gas well treatment environments. The integration of experimental measurements, adsorption modeling, kinetic and thermodynamic analyses, thermal characterization, and statistical optimization provided detailed insight into the inhibition mechanism and operational behavior of the extract. Weight loss measurements demonstrated that MSLE exhibits strong concentration-dependent inhibition efficiency within the investigated temperature range of 323–343 K and immersion times of 2–10 h. The inhibition efficiency increased with increasing inhibitor concentration, reaching a maximum experimental value of 98.40 % at 0.8 g/L, 333 K, and 2 h. Although a slight reduction in efficiency was observed at prolonged immersion times and higher temperatures, significant protection was maintained, indicating the formation of a stable protective film under elevated temperature conditions.

Adsorption studies revealed excellent agreement between the experimental data and the Langmuir, Freundlich, and Temkin isotherm models ($R^2 = 0.942\text{--}0.995$), confirming that the inhibition mechanism is governed by adsorption of phytochemical constituents onto the steel surface. The negative values of Gibbs free energy of adsorption (ΔG_{ads}) confirmed spontaneous adsorption, while the magnitude of the thermodynamic parameters suggests a predominantly physisorption-controlled process with possible contributions from chemisorption through heteroatom coordination. The consistency among multiple isotherm models further indicates that the steel surface behaves as a heterogeneous adsorption system influenced by lateral interactions among adsorbed species. Kinetic evaluation using Arrhenius and transition state analyses showed that the apparent activation energy increased in the presence of MSLE compared to the uninhibited system,

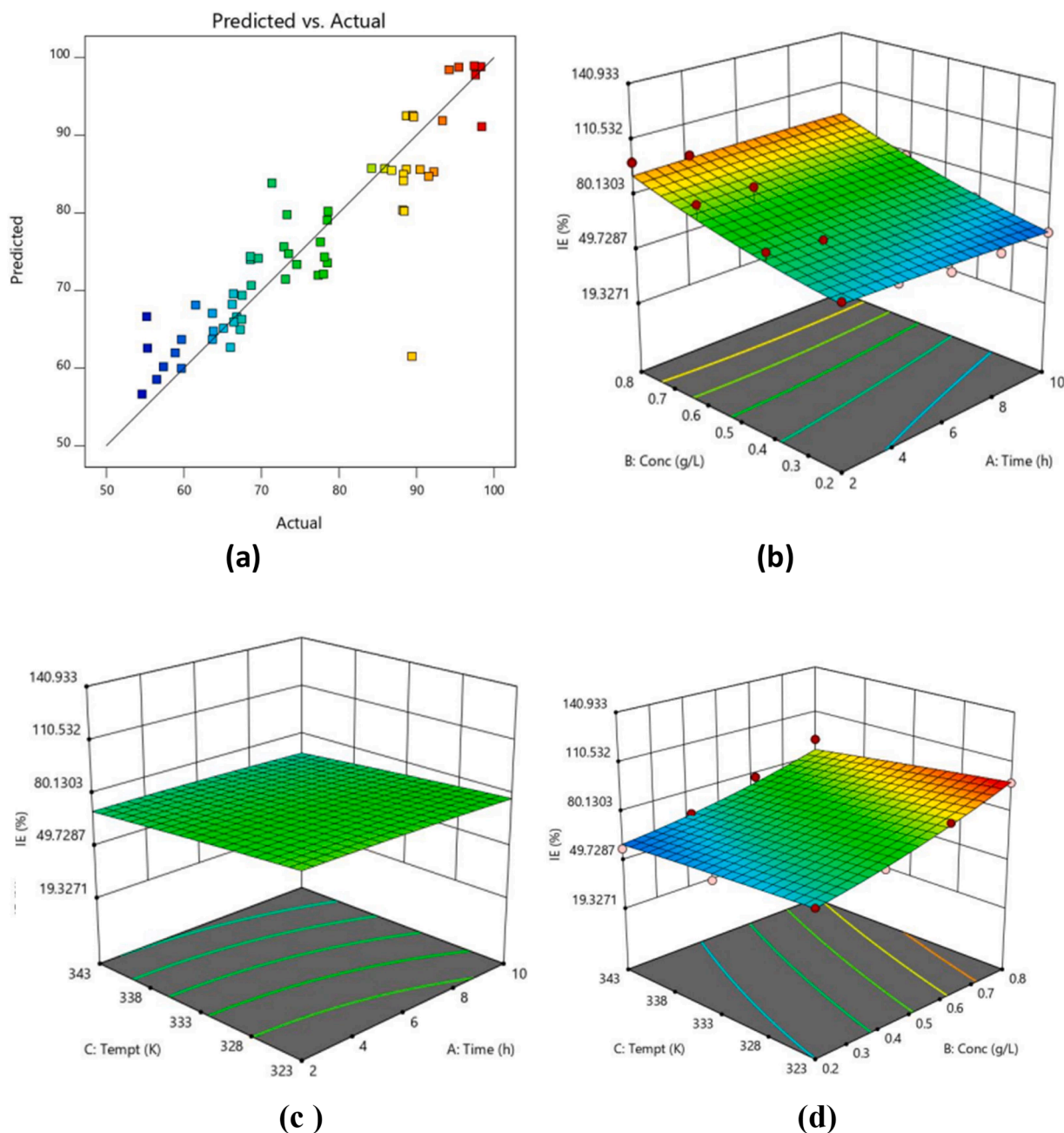


Fig. 8. (a) RSM experimental vs predicted IE (b) 3D RSM diagnostic plot of IE versus inhibitor concentration and time (c) RSM diagnostic plot of IE versus temperature and time d RSM diagnostic plot of IE versus temperature and inhibitor concentration.

Table 5
Validation of optimal parameters.

System	Optimal inhibition Conc.g/L)	Optimal temperature (K)	Optimal time(h)	Optimal inhibitor efficiency
X65 steel in 1 M HCl with MSLE	0.8	343	9.1252	90.24

demonstrating that the extract raises the energy barrier of the corrosion reaction. This behavior is characteristic of adsorption-mediated surface blocking. The positive enthalpy of activation values confirmed the endothermic nature of the corrosion process, while negative entropy values indicated increased order at the metal–solution interface due to

formation of the activated complex and structured inhibitor film. Collectively, these findings support an adsorption-driven inhibition mechanism that suppresses charge transfer reactions.

Thermogravimetric and differential thermal analyses revealed negligible mass loss below 120 °C, corresponding to moisture removal, and significant decomposition between 250 and 380 °C attributed to degradation of active phytochemical constituents. The thermal stability profile confirms that MSLE possesses sufficient thermal resilience for laboratory corrosion testing and practical oilfield acidizing operations within the investigated temperature range. Response Surface Methodology analysis identified inhibitor concentration as the most significant factor affecting inhibition efficiency ($p < 0.0001$), followed by temperature, while immersion time exhibited relatively minor independent effects within the studied domain. The developed quadratic model was statistically significant and demonstrated satisfactory predictive

capability ($R^2 = 0.8098$, Adjusted $R^2 = 0.7755$, Predicted $R^2 = 0.7123$, Adeq Precision = 16.76). Numerical optimization predicted optimal operating conditions of 0.8 g/L, 343 K, and 9.13 h, with a validated inhibition efficiency of 90.24 %, confirming the reliability of the statistical model for process optimization and design space exploration. The findings establish *Mitragyna speciosa* leaf extract as a thermodynamically feasible, thermally stable, and statistically optimizable green corrosion inhibitor for X65 steel in acidic media. The integrated thermodynamic-kinetic-thermal-statistical framework adopted in this study provides deeper mechanistic understanding compared to conventional efficiency-based assessments and offers a stronger scientific foundation for the deployment of plant-derived inhibitors in high-temperature oilfield systems. These results contribute to the advancement of sustainable corrosion mitigation strategies by demonstrating that naturally derived phytochemical systems can achieve high protection efficiency while maintaining environmental compatibility and potential scalability for industrial applications.

CRediT authorship contribution statement

Silas.O. Okuma: Conceptualization, Methodology, Investigation, Writing – review & editing. **Cynthia C. Nwaeju-Okechukwu:** Formal analysis, Writing – review & editing. **Benjamin U. Oreko:** Writing – review & editing, 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.

Data availability

Data will be made available on request.

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