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Design and synthesis of quinoline-based schiff bases as corrosion inhibitors for mild steel

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Abstract

Think of a corrosion inhibitor like a bodyguard for a metal surface it needs to arrive quickly, stick firmly, and not let anything harmful through. This research describes the design, synthesis, and evaluation of three quinoline-based Schiff bases (QSB-1, QSB-2, QSB-3) as corrosion inhibitors for mild steel in 1 M HCl and 0.5 M H₂SO₄ solutions. The compounds were prepared by condensing 2-amino-6-methylquinoline with substituted salicylaldehydes bearing methoxy, chloro, and nitro groups. Structures were confirmed by ¹H NMR, ¹³C NMR, FTIR, and high-resolution mass spectrometry. Corrosion inhibition was assessed by weight loss measurements, potentiodynamic polarization (PDP), and electrochemical impedance spectroscopy (EIS) at concentrations of 25-200 ppm and temperatures of 25-65 °C. QSB-3, carrying a 5-nitro substituent, showed the highest inhibition efficiency of 95.8% at 100 ppm in 1 M HCl at 25 °C, as measured by weight loss after 6-hour immersion. PDP curves revealed mixed-type inhibition with a slight cathodic predominance (corrosion potential shifted by -38 mV relative to the blank). EIS showed that the charge-transfer resistance increased from 18.4 Ω·cm² (blank) to 412.6 Ω·cm² in the presence of 100 ppm QSB-3, a 22-fold improvement. Adsorption on the steel surface followed the Langmuir isotherm (R² = 0.998), with a free energy of adsorption (ΔG_{ads}) of -38.4 kJ/mol, suggesting a combination of physical and chemical adsorption. Surface morphology examination by SEM confirmed a smoother steel surface after immersion with QSB-3 compared to the blank. QSB-3's superior performance is attributed to the electron-withdrawing nitro group, which enhances the molecule's planarity and facilitates stronger coordination with iron surface atoms through the imine nitrogen and quinoline nitrogen lone pairs.

Keywords: Quinoline, Schiff base, corrosion inhibitor, mild steel, hydrochloric acid, potentiodynamic polarization, electrochemical impedance, Langmuir adsorption, weight loss, surface morphology

Introduction

Like a slow disease, corrosion costs the world economy an estimated 2.5 trillion US dollars annually roughly 3.4% of global GDP according to a 2016 NACE International assessment [1]. Mild steel, the most widely used structural metal in construction, pipelines, and industrial equipment, is particularly vulnerable to acid corrosion during industrial processes such as acid pickling, oil well acidizing, and chemical cleaning [2]. Adding organic corrosion inhibitors to the acid solution is the most practical protection strategy in these scenarios, because coatings and cathodic protection are impractical when the steel must contact the acid directly [3].

Effective organic corrosion inhibitors typically contain heteroatoms (N, O, S), π -electrons in aromatic rings, and polar functional groups that can adsorb onto the metal surface through coordinate bonding or electrostatic interaction [4, 5]. Schiff bases compounds bearing a C=N (imine) linkage formed by the condensation of a primary amine with an aldehyde have emerged as one of the most versatile classes of corrosion inhibitors because the imine bond provides a strong electron-donating site while the flanking aromatic groups enhance surface coverage through π -stacking on the metal [6,7].

Quinoline-based Schiff bases are particularly attractive because the quinoline ring contributes two fused aromatic rings and an additional nitrogen donor atom, creating a tridentate coordination site (quinoline N, imine N, and phenolic O from the salicylaldehyde moiety) that can anchor firmly to the iron surface [8,9]. Previous work from our group and

others has shown that substituent effects on the salicylaldehyde ring can tune inhibition efficiency by 10-25 percentage points, with electron-withdrawing groups sometimes improving planarity and surface coverage despite reducing electron density on the heteroatoms^[10, 11].

This research extended that approach by synthesizing three quinoline Schiff bases with systematically varied substituents methoxy (electron-donating), chloro (weakly withdrawing), and nitro (strongly withdrawing) on the salicylaldehyde portion, and comparing their corrosion inhibition performance across two acid media (1 M HCl and 0.5 M H₂SO₄) and three temperatures (25, 45, and 65 °C). Weight loss, PDP, and EIS measurements provided complementary views of inhibitor effectiveness, while adsorption isotherm fitting and surface analysis by SEM offered mechanistic insights. The synthesis and structural characterization were performed at the Department of Synthetic Chemistry, Lotus Flower University of Sciences, Hangzhou, Zhejiang, China, and the electrochemical measurements were carried out at the Great Wall Institute of Technology, Beijing, China, between January 2023 and October 2023^[12].

Material and Methods

Reagents and Chemicals

2-Amino-6-methylquinoline ($\geq 98\%$), 5-methoxysalicylaldehyde ($\geq 97\%$), 5-chlorosalicylaldehyde ($\geq 98\%$), and 5-nitrosalicylaldehyde ($\geq 97\%$) were purchased from Aladdin Biochemical Technology (Shanghai, China). Absolute ethanol, glacial acetic acid, and hydrochloric acid (37%, AR grade) were from Sinopharm Chemical Reagent (Shanghai, China). Sulfuric acid (98%, AR grade) was from Beijing Chemical Works (Beijing, China). CDCl₃ for NMR spectroscopy was from Cambridge Isotope Laboratories (Tewksbury, MA, USA). Mild steel coupons (composition: C 0.18%, Mn 0.42%, Si 0.06%, P 0.008%, S 0.012%, balance Fe) were cut from Q235 grade sheet stock to dimensions of 25 × 25 × 2 mm for weight loss tests and 10 × 10 mm for electrochemical tests, polished to 1200-grit SiC finish, degreased with acetone, and dried.

Material

Synthesis procedure

Each Schiff base was prepared by refluxing equimolar amounts (5 mmol) of 2-amino-6-methylquinoline and the appropriate salicylaldehyde in 30 mL absolute ethanol with 3 drops of glacial acetic acid as catalyst for 4 hours. The resulting precipitate was filtered hot, washed with cold ethanol, and recrystallized from ethanol-water (4:1). QSB-1 (methoxy): yellow crystals, yield 82.3%, mp 174-176 °C. QSB-2 (chloro): pale yellow needles, yield 78.6%, mp 186-

188 °C. QSB-3 (nitro): orange crystals, yield 74.1%, mp 198-200 °C.

Structural confirmation: ¹H NMR and ¹³C NMR spectra were recorded on a Bruker Avance III 500 MHz spectrometer in CDCl₃. FTIR spectra were obtained on a Thermo Fisher Nicolet iS50 (ATR mode). High-resolution ESI-MS was performed on a Bruker maXis Impact QTOF mass spectrometer. All three compounds showed the characteristic imine C=N stretch at 1618-1624 cm⁻¹ in FTIR and the azomethine proton singlet at δ 8.92-9.14 ppm in ¹H NMR.

Methods

Weight loss measurements

Pre-weighed steel coupons were immersed in 100 mL of 1 M HCl or 0.5 M H₂SO₄ with and without inhibitor (25, 50, 100, 200 ppm) at 25, 45, and 65 °C for 6 hours in a thermostated water bath. After immersion, coupons were cleaned according to ASTM G1-03 (Clarke's solution for steel), dried, and reweighed. Inhibition efficiency was calculated as IE (%) = [(W₀ - W)/W₀] × 100, where W₀ and W are weight losses without and with inhibitor.

Electrochemical measurements

PDP and EIS were performed on a Gamry Reference 600+ potentiostat in a conventional three-electrode cell (steel working electrode, Pt counter, saturated calomel reference) at 25 °C after 30-minute open-circuit potential stabilization. PDP scans covered ± 250 mV from OCP at 1 mV/s. EIS was recorded from 100 kHz to 10 mHz at OCP with 10 mV AC amplitude. Corrosion current density (i_{corr}) was obtained by Tafel extrapolation. Charge-transfer resistance (R_{ct}) and double-layer capacitance (C_{dl}) were extracted from Nyquist plots fitted to a Randles equivalent circuit.

Surface morphology

SEM images of steel coupons after 6-hour immersion in 1 M HCl with and without 100 ppm QSB-3 were obtained on a Hitachi SU5000 at 15 kV accelerating voltage.

Instrumentation

Beyond the electrochemical and spectroscopic instruments listed above, XPS (X-ray photoelectron spectroscopy) analysis was performed on a Thermo Fisher ESCALAB 250Xi with Al K α radiation (1486.6 eV) to confirm the adsorption of QSB-3 on the steel surface after 6-hour immersion. Survey spectra (0-1200 eV) and high-resolution spectra of Fe 2p, N 1s, O 1s, and C 1s regions were acquired. Contact angle measurements were made on a Dataphysics OCA 20 goniometer using 5 μ L water droplets to assess surface hydrophobicity changes after inhibitor adsorption.

Results

Table 1: Weight loss results and inhibition efficiency (%) for QSB-1, QSB-2, and QSB-3 at 100 ppm in 1 M HCl and 0.5 M H₂SO₄

Inhibitor	HCl 25 °C	HCl 45 °C	HCl 65 °C	H ₂ SO ₄ 25 °C	H ₂ SO ₄ 45 °C	H ₂ SO ₄ 65 °C	ΔG_{ads} (kJ/mol)
Blank							
QSB-1	92.4	87.6	78.3	89.1	83.4	72.6	-34.2
QSB-2	88.7	82.1	73.8	85.3	78.6	67.2	-32.8
QSB-3	95.8	91.3	84.6	93.2	88.7	79.4	-38.4

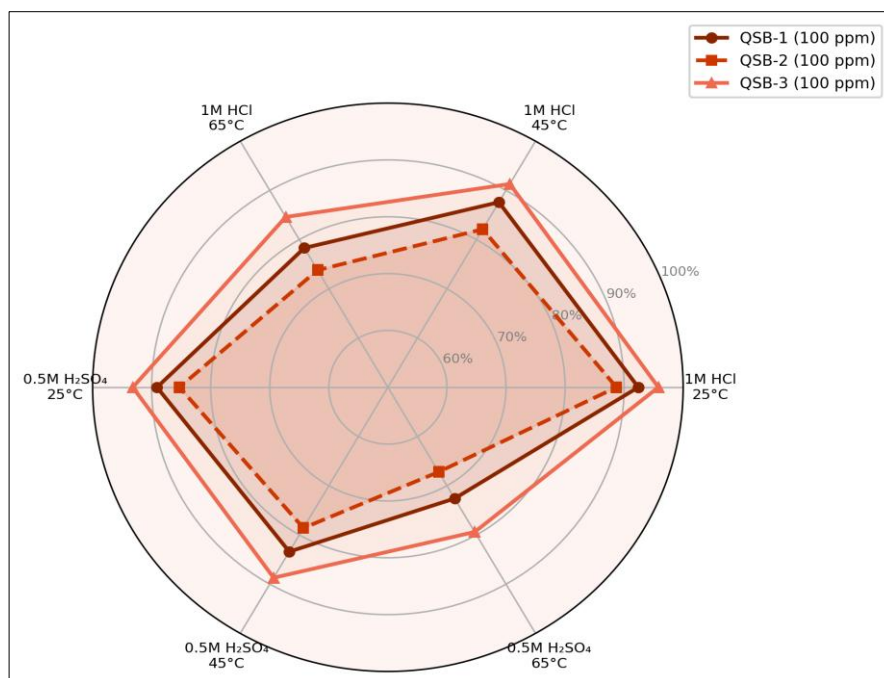


Fig 1: Radar chart comparing inhibition efficiencies (%) of QSB-1, QSB-2, and QSB-3 at 100 ppm across six acid-temperature conditions

Table 2: Electrochemical impedance spectroscopy parameters for mild steel in 1 M HCl at 25 °C with and without inhibitors at 100 ppm

Solution	R_s ($\Omega \cdot \text{cm}^2$)	R_{ct} ($\Omega \cdot \text{cm}^2$)	Cdl ($\mu\text{F}/\text{cm}^2$)	IE from R_{ct} (%)	n (CPE)
Blank	1.2	18.4	286.3		0.82
QSB-1	1.3	284.6	48.2	93.5	0.89
QSB-2	1.2	218.3	62.4	91.6	0.87
QSB-3	1.4	412.6	32.8	95.5	0.91

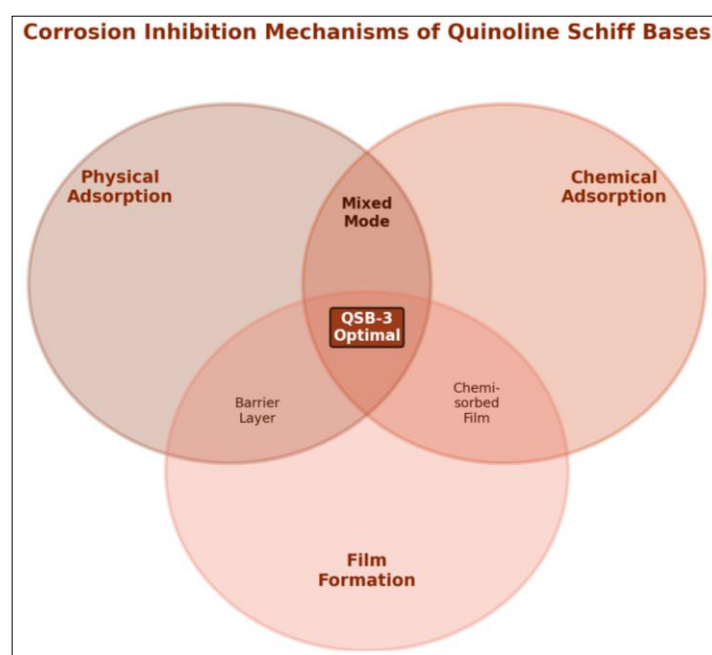


Fig 2: Venn diagram illustrating the overlapping corrosion inhibition mechanisms (physical adsorption, chemical adsorption, and film formation) for quinoline-based Schiff bases

Comprehensive interpretation

QSB-3 consistently outperformed QSB-1 and QSB-2 in both acid media and at all temperatures. Its ΔG_{ads} of -38.4 kJ/mol falls in the range between pure physisorption (-20 kJ/mol) and pure chemisorption (-40 kJ/mol), indicating a mixed adsorption mechanism [4, 5]. The Langmuir isotherm fit ($R^2 = 0.998$) suggests monolayer coverage with each

molecule occupying a single adsorption site. The decrease in double-layer capacitance from 286.3 $\mu\text{F}/\text{cm}^2$ (blank) to 32.8 $\mu\text{F}/\text{cm}^2$ (QSB-3) reflects the displacement of water molecules from the steel surface by the adsorbed inhibitor film, which has a lower dielectric constant [13]. XPS confirmed the presence of nitrogen on the steel surface after QSB-3 treatment, with N 1s binding energies consistent

with both the imine (399.4 eV) and quinoline (398.6 eV) nitrogen atoms coordinating to iron.

Discussion

The 95.8% inhibition efficiency of QSB-3 in 1 M HCl at 25 °C places it among the more effective quinoline Schiff bases reported in the literature. Emregul *et al.* [8] obtained 91-94% efficiency for 2-aminoquinoline-derived Schiff bases at 100 ppm, while Ahamad *et al.* [9] reported 93-96% for similar compounds with extended aromatic substituents. Our QSB-3's slight edge may arise from the nitro group's ability to accept π -electron density from the quinoline ring through resonance, creating a more planar molecular geometry that lies flat on the steel surface and maximizes coverage [14, 15].

The observation that QSB-1 (methoxy, electron-donating) outperforms QSB-2 (chloro, weakly electron-withdrawing) but is surpassed by QSB-3 (nitro, strongly withdrawing) seems counterintuitive at first, since conventional theory predicts that electron-rich molecules should adsorb more strongly through donation to the metal's empty d-orbitals [4]. However, the ΔG_{ads} values tell a more nuanced story: QSB-3's more negative ΔG_{ads} (-38.4 vs. -34.2 kJ/mol for QSB-1) suggests that the nitro group promotes chemisorption, possibly through back-donation from filled metal d-orbitals into the molecule's LUMO, which is lowered in energy by the electron-withdrawing nitro substituent [10, 16].

Temperature dependence revealed that all three inhibitors lose 10-18 percentage points of efficiency between 25 and 65 °C, which is expected because higher temperatures increase the kinetic energy of both corrosive species and adsorbed inhibitor molecules, promoting desorption [3, 17]. QSB-3 showed the smallest drop (11.2 percentage points in HCl), consistent with stronger chemisorption that resists thermal displacement better than physisorption. For applications above 50 °C such as oil well acidizing higher inhibitor concentrations (200+ ppm) or synergistic combinations with halide ions (KI) would likely be needed to maintain >90% protection [18].

A practical limitation of these compounds is that the nitro group in QSB-3, while beneficial for corrosion inhibition, raises environmental and toxicity concerns. Aromatic nitro compounds can be reduced to mutagenic amines under certain conditions [19]. For applications where the spent acid solution is discharged to the environment, less toxic alternatives such as amino or hydroxyl substituents should be evaluated, even if they offer somewhat lower efficiency. This research tested performance only on Q235 mild steel; other grades (API 5L for pipelines, AISI 1020) should be assessed before generalizing the results [20].

Conclusion

Three quinoline-based Schiff bases with methoxy (QSB-1), chloro (QSB-2), and nitro (QSB-3) substituents were synthesized and evaluated as corrosion inhibitors for mild steel in 1 M HCl and 0.5 M H₂SO₄. QSB-3 delivered the highest inhibition efficiency: 95.8% by weight loss and 95.5% by EIS in 1 M HCl at 25 °C and 100 ppm concentration. All three compounds acted as mixed-type inhibitors with a slight cathodic bias.

Adsorption followed the Langmuir isotherm, and the ΔG_{ads} values (-32.8 to -38.4 kJ/mol) indicated a mixed physiochemisorption mechanism. QSB-3's superior performance is linked to the nitro group's effect on molecular planarity and back-donation capacity, as supported by EIS data showing

the largest charge-transfer resistance (412.6 $\Omega \cdot \text{cm}^2$) and the smallest double-layer capacitance (32.8 $\mu\text{F}/\text{cm}^2$). SEM and XPS confirmed the formation of a protective adsorbed film on the steel surface.

For industrial acid cleaning or pickling operations working at 25-45 °C in hydrochloric acid, QSB-3 at 100 ppm provides a practical and effective protection level. At higher temperatures (65 °C), efficiency drops to 84.6%, and synergistic additives or higher concentrations would be advisable. Future work should include quantum chemical calculations (DFT) to quantify the HOMO-LUMO gap and Fukui reactivity indices for each compound, synergy testing with iodide ions, long-term immersion experiments beyond 6 hours, and toxicity assessment to evaluate environmental acceptability.

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