

Original Research

Effect of Sulfide Ions on the Inhibition Performance and Mechanism of Metal–Organic Frameworks for Copper in Marine Environments

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Abstract

Background: Metal–organic framework (MOF)-based corrosion inhibitors are susceptible to interference from environmental ions in practical applications; however, the underlying failure mechanisms are poorly understood. In particular, the influence of sulfide ions (S^{2-}) on the ability of MOFs to prevent copper corrosion has not been systematically investigated. **Methods:** Three isostructural MOFs—Zn-zeolitic imidazolate framework (ZIF), Co-ZIF, and Ni-ZIF—were synthesized and applied as copper corrosion inhibitors. Their protective performance was systematically evaluated in sulfide-containing environments using electrochemical techniques. The structural evolution of the MOFs upon sulfide exposure was characterized to elucidate the mechanism of inhibition failure. **Results:** All three materials efficiently inhibited corrosion at a sulfide ion concentration of 50 mg/L, with inhibition efficiencies reaching 90.06% (Zn-ZIF), 93.39% (Co-ZIF), and 89.31% (Ni-ZIF). However, when the sodium sulfide nonahydrate ($Na_2S \cdot 9H_2O$) concentration was increased to 10 mg/L, the inhibition efficiencies dropped to 15.54%, 18.11%, and 4.00%, respectively. Mechanistic analysis uncovered a novel failure pathway, namely competitive coordination between sulfide ions and MOF metal centers, which induces structural collapse of the framework and consequently destroys the protective film previously formed on the copper surface. **Conclusions:** This study reveals a quantitative relationship between sulfide ion concentration and the performance of MOF-based corrosion inhibitors, defines the applicability boundaries of MOF-based inhibitors under complex sulfur-containing service conditions, and provides critical practical guidance for the deployment of such inhibitors while elucidating a fundamental failure mechanism driven by competitive coordination.

Keywords: MOFs; sulfide ions; copper; corrosion protection performance; structure–activity relationship

1. Introduction

Metal materials possess excellent thermal and electrical conductivity as well as ductility, making them indispensable in sectors such as automotive manufacturing, marine engineering, and microelectronics [1,2,3]. However, the escalating exploitation of marine resources, driven by ongoing technological advances, has rendered marine environmental corrosion a critical and ubiquitous threat to offshore infrastructure and assets. High humidity and corrosive media readily induce chemical reactions on metal surfaces, leading to significant material degradation [4,5,6]. This corrosion attack not only severely compromises the structural integrity and service longevity of marine equipment but also precipitates exorbitant economic burdens and unmitigated environmental hazards [7,8,9]. Among diverse methods, including coating protection and electrochemical protection [10], corrosion inhibitors are extensively employed owing to their cost-effectiveness and operational simplicity [11,12,13]. Corrosion inhibitors protect metal substrates through electron transfer and strong surface interactions. Extensive research highlights that organic compounds containing unsaturated bonds or heteroatoms, such as nitrogen, oxygen, and sulfur, exhibit superior inhibition properties [14,15,16]. While traditional inhibitors are effective,

they often face challenges related to biological toxicity and poor dispersion within coatings. Introducing metal-organic frameworks (MOFs) to tackle these limitations and improve protective performance. MOFs are widely utilized in sensing, semiconductors, drug delivery, catalysis, and other fields due to their advantages such as tunable structure, porosity, and high specific surface area [17,18,19]. In recent years, MOFs have emerged as a promising alternative in the corrosion field [20,21,22,23]. MOF materials can act as corrosion inhibitors. Firstly, this is attributed to their intrinsic functional groups and heterocyclic structures. Secondly, they can function as carriers for corrosion inhibitors because of their large specific surface area, tunable structure, and unsaturated coordination sites. For example, Ashika et al. [24] investigated the copper-based metal–organic framework Hong Kong University of Science and Technology-1 (HKUST-1) as a corrosion inhibitor for AISI 5140 steel in a 3.5 wt% NaCl solution across a temperature range of 30 °C to 50 °C. Their findings confirmed exceptional temperature-dependent performance, with the lowest corrosion rate of 0.249 mm y^{−1} and maximum inhibition efficiency of 88.9% achieved at 50 °C using a concentration of 0.005 g L^{−1}. These results highlight HKUST-1's potential as a promising environmentally benign cor-



rosion inhibitor for saline environments. Besides, Han et al. [25] developed UiO-67-Ce with controlled loading of benzotriazole (BTA). The UiO-67-Ce@BTA/epoxy coating exhibits a high corrosion inhibition efficiency of 99.95%, which is attributed to the efficient release of the bifunctional inhibitors.

However, the efficiency of these inhibitors is highly dependent on environmental variables, including temperature, pH, and the presence of complexing ions. For example, inhibitors containing N, O, or S atoms are prone to protonation in acidic environments, which alters their electron-donating ability and adsorption behavior [26,27,28]. Furthermore, the presence of ions with strong complexing abilities, such as sulfide ions (S^{2-}) and NH_4^+ , can interfere with the inhibitor's adsorption. For example, Ruonan Tang et al. [29] developed S^{2-} -responsive metronidazole@zeolitic imidazolate framework (ZIF)-8 nanocontainers with high drug loading (59.8 mg/g). Release reached 90.8% at 5 mM S^{2-} via non-Fickian diffusion. At 300 $\mu\text{g mL}^{-1}$, the nanocontainers achieved 100% antibacterial efficacy against sulfate-reducing bacteria and eliminated corrosion pits on Q235 steel.

MOFs are organic-inorganic hybrids characterized by high porosity and tunable frameworks [30,31,32]. These features allow MOFs to serve either as active inhibitors leveraging their abundant heterocyclic functional groups—or as intelligent nanocarriers for the controlled release of inhibitor molecules. Despite their potential, the structural stability and inhibition mechanisms of various MOF configurations in aggressive ion-rich environments remain insufficiently understood. This paper explores the structural stability and inhibitory performance of MOFs within a sulfide ion corrosion environment, simulated by adding sodium sulfide nonahydrate to a neutral medium. By utilizing electrochemical testing methods complemented by scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), and X-ray diffraction (XRD) characterization, this study aims to reveal how specific metal centers and corrosive ions influence the protective efficacy of MOF-based systems.

2. Materials and Methods

2.1 Experimental Materials

The metallic material used in the experiment was T2 copper, purchased from Shandong Senke Company in China. The chemical reagents employed included: zinc nitrate hexahydrate, cobalt nitrate hexahydrate, and nickel nitrate hexahydrate, obtained from Sigma-Aldrich; 2-methylimidazole, methanol, dimethylformamide, sodium chloride, and sodium sulfide nonahydrate ($Na_2S \cdot 9H_2O$), obtained from Aladdin. All reagents were of analytical grade and were not further purified. The sodium chloride solution used for testing was prepared by mixing analytical-grade solid sodium chloride with deionized water. The solvents used were methanol and ammonia, both of which

were of analytical grade and did not require further purification.

2.2 Experimental Procedure

The synthesis of ZIF-8 derivatives was carried out via the hydrothermal method, as detailed below [33,34]: First, 2.0 mmol of 2-methylimidazole and 2.0 mmol zinc nitrate were dissolved in 80 mL of dimethylformamide and subjected to ultrasonic treatment; the resulting mixture was then transferred to a hydrothermal reactor and heated at 140 °C for 24 h. After natural cooling of the reaction system, the white product was obtained by centrifugation, washed with methanol, and dried overnight in a vacuum drying oven to yield Zn-ZIF. Using the same molar ratios, the metal sources were replaced with cobalt nitrate hexahydrate and nickel nitrate hexahydrate, respectively, with all other steps remaining unchanged; this procedure yielded purple Co-ZIF and yellow Ni-ZIF, respectively.

2.3 Characterization and Methods

The synthesized materials were characterized using SEM (NovaNano SEM 450, FEI, Eindhoven, Netherlands), Transmission Electron Microscopy (TEM) (Tecnai G2 F30 TEM, FEI, Eindhoven, Netherlands), and Infrared Spectroscopy (VERTEX 70 FT-IR, Bruker, Ettlingen, Germany). SEM and TEM were utilized to examine the surface morphology and microstructure, with TEM providing higher-resolution imaging of intricate structural details. Infrared Spectroscopy was used to identify the molecular structure and chemical bonding by analyzing characteristic absorption wavelengths. These structural and chemical features directly influence the material's interaction with the substrate, thereby determining its protective and electrochemical performance.

Electrochemical tests were conducted on the treated metal surfaces using an electrochemical workstation (Wuhan CS310, Wuhan, Hubei, China). Open-circuit potential measurements were used to assess the equilibrium electrochemical state. Electrochemical impedance spectroscopy (EIS) analyzed charge transfer and mass transfer processes at the electrode interface. Potentiodynamic polarization was employed to investigate the current-time response at a fixed potential, providing data on electrochemical stability and corrosion inhibition. These analyses demonstrate that the synthesized materials significantly alter the electrochemical properties of the metal surfaces. Additionally, based on the acquired corrosion current density (i_{corr}) values, the inhibition efficiency (η) derived from polarization curves can be computed using Eqn. 1 [35,36]:

$$\eta(\%) = \frac{i_{corr}^0 - i_{corr}}{i_{corr}^0} \times 100\% \quad (1)$$

Table 1. Element content analysis of Na₂S·9H₂O treated different metal-sources-ZIFs.

Samples	C (wt.%)	O (wt.%)	S (wt.%)	Zn (wt.%)	Co (wt.%)	Ni (wt.%)
Zn-ZIF	54.52	30.87	3.58	11.03	/	/
Co-ZIF	47.53	30.59	9.96	/	11.92	/
Ni-ZIF	50.07	26.17	10	/	/	13.76

where i_{corr} and i_{corr}^0 denoted the corrosion current density acquired in the presence and absence of the inhibitor, respectively.

To ensure the reproducibility of experimental results, this study conducted three parallel measurements for each experimental group.

3. Results

Zn-ZIF, Co-ZIF, and Ni-ZIF were immersed in a 0.5 mol/L NaCl solution containing 10 mg/L Na₂S·9H₂O to evaluate their structural stability. The microscopic morphologies of these three ZIFs with different metal centers were characterized using SEM and TEM. According to the SEM images (Fig. 1a–c), the pristine ZIFs exhibited consistent polyhedral shapes and relatively intact particles. However, after immersion in the Na₂S·9H₂O solution (Fig. 1d–f), the original structures of Zn-ZIF, Co-ZIF, and Ni-ZIF were compromised, resulting in varying degrees of fragmentation. Specifically, the well-defined geometric forms of these ZIFs were lost as the particles broke down into irregular fragments.

Detailed structural transformations were further examined through TEM analysis. The untreated ZIFs (Fig. 2a–c) were uniformly sized, regularly shaped, and possessed well-defined boundaries. In the presence of Na₂S·9H₂O (Fig. 2d–f), the morphology of each ZIF changed significantly: the edges of Zn-ZIF became indistinct and exhibited a hooking phenomenon; Co-ZIF was essentially fragmented, exhibiting cracks and fractures; and Ni-ZIF similarly showed evidence of rupture and structural disintegration. Furthermore, Energy Dispersive X-ray Spectroscopy (EDS) elemental analysis was performed on these ZIFs, with the results summarized in Table 1. The analysis detected the presence of sulfur in all three ZIFs after Na₂S·9H₂O treatment, with Zn-ZIF, Co-ZIF, and Ni-ZIF containing 3.58%, 9.96%, and 10% sulfur, respectively. These results indicate that Na₂S·9H₂O treatment leads to the presence of sulfides in ZIFs, which potentially induces alterations in their structure and properties.

The XRD patterns of the three ZIFs with different metal centers were illustrated in Fig. 3. XRD patterns were obtained using X'Pert 3 powder XRD-1 (PANalytical B.V., Almelo, Netherlands). Zn-ZIF, Co-ZIF, and Ni-ZIF all exhibit diffraction patterns consistent with those previously reported [37]. Rietveld analysis of XRD patterns showed characteristic Bragg peaks confirming formation of single phase cubic structure with an average particle size. Following treatment with Na₂S·9H₂O, distinct changes occurred

in the crystalline structures of each material: for Zn-ZIF, new peaks emerged at $2\theta = 28.6^\circ$, 47.6° , and 56.3° , corresponding to ZnS [38,39]; CoS signals appeared at 35.08° and 46.6° [40], and NiS features were observed at 30.1° and 45.0° [41]. The appearance of these new reflections, which precisely match the standard diffraction data for the respective sulfides, confirms that the metal ions within the ZIF frameworks reacted with S²⁻ in the solution to successfully generate the corresponding metal sulfide phases.

3.1 Evaluation of the Effect of Sulfide Ions on the Corrosion Inhibition Performance of ZIFs

3.1.1 Electrochemical Test and Results

The corrosion behavior of copper in a 0.5 mol/L NaCl solution containing 10 mg/L sodium sulfide was evaluated by electrochemical test method, as shown in Fig. 4, in the polarization curve (Fig. 4a), when S²⁻ was added, the corrosion potential moved in a small positive direction, and the corrosion current density hardly changed. It shows that its effect on the corrosion behavior of copper metal is not obvious. From the Nyquist plot (Fig. 4b), it was found that the diameter of the capacitive arc changed only slightly when S²⁻ was present, indicating that the effect of S²⁻ on the corrosive behavior of copper metals was negligible in this study.

Fig. 5 showed the polarization curves of a copper electrode immersed in 0.5 mol/L NaCl solution containing three ZIFs in an environment containing Na₂S·9H₂O. First, the polarization curves of copper in 0.5 mol/L NaCl solution containing ZIFs with different metal sources were measured, and the fitting results of the polarization curves were presented in Table 2, which included the corrosion current density (i_{corr}), corrosion potential (E_{corr}), and anodic and cathodic Tafel slopes. The i_{corr} value can be used to evaluate the corrosion inhibition performance of an inhibitor [42]. In a corrosive environment, the corrosion current density of a material is positively correlated with the degree of damage. Therefore, a high current density indicates a severe corrosion condition, whereas a low current density signifies a lesser degree of damage. The polarization curves show that upon the addition of ZIFs, i_{corr} decreased significantly, and this effect was evident in both the cathodic and anodic branches. This indicates that the three as-prepared ZIFs possess excellent inhibitory effects on both copper dissolution and oxygen reduction. As can be seen from Fig. 5a, compared with the blank group, when Zn-ZIF was added at a concentration of 50 mg/L, the E_{corr} of copper shifted positively while i_{corr} decreased, with a significant drop in i_{corr} .

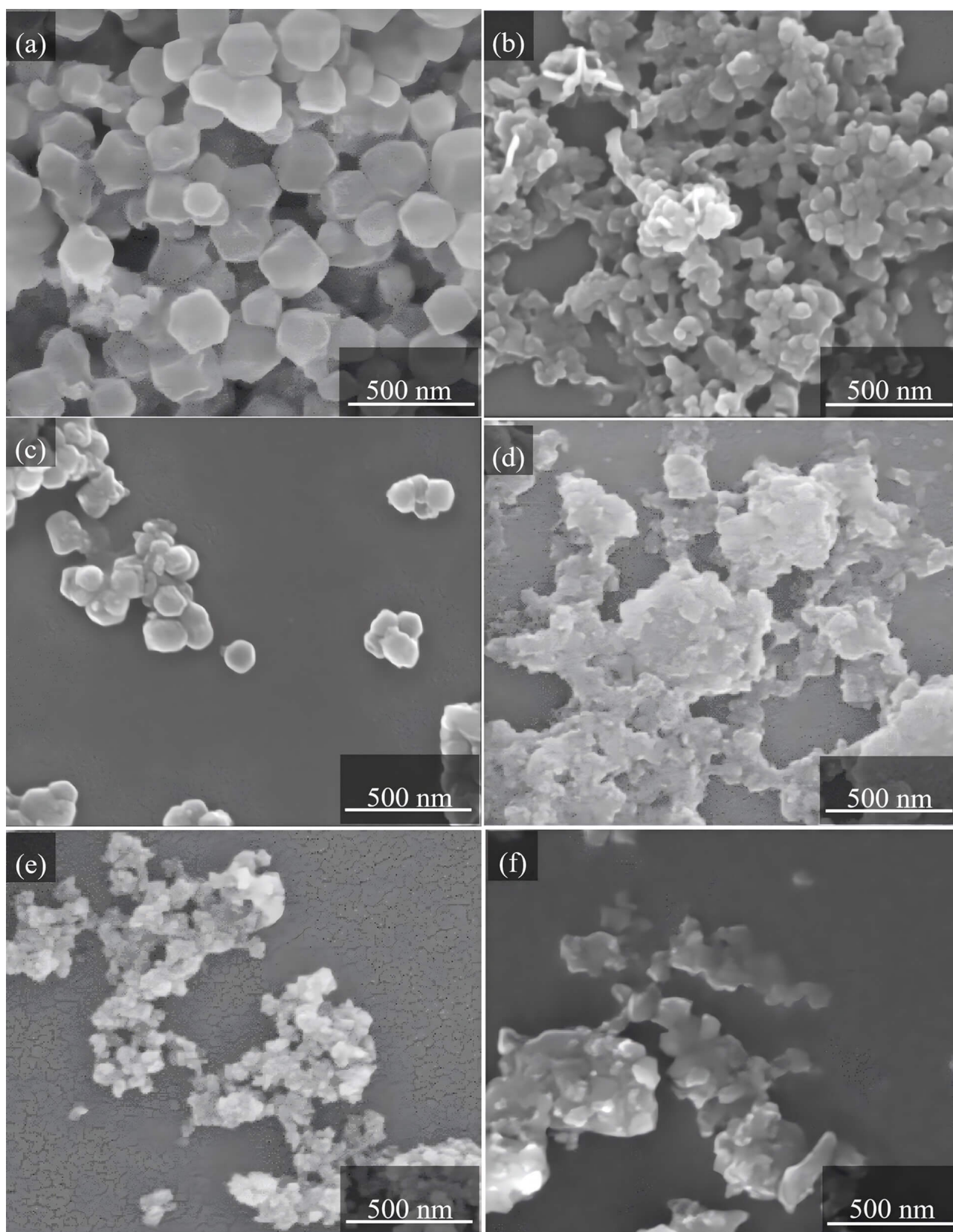


Fig. 1. Scanning electron microscopy (SEM) images before and after 0.5 mol/L NaCl solution containing 10 mg/L $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ treatment at 25 °C. (a) Zn-ZIF. (b) Co-ZIF. (c) Ni-ZIF. (d) Zn-ZIF ($\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$). (e) Co-ZIF ($\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$). (f) Ni-ZIF ($\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$). ZIF, zeolitic imidazolate framework. Scale bar = 500 nm.

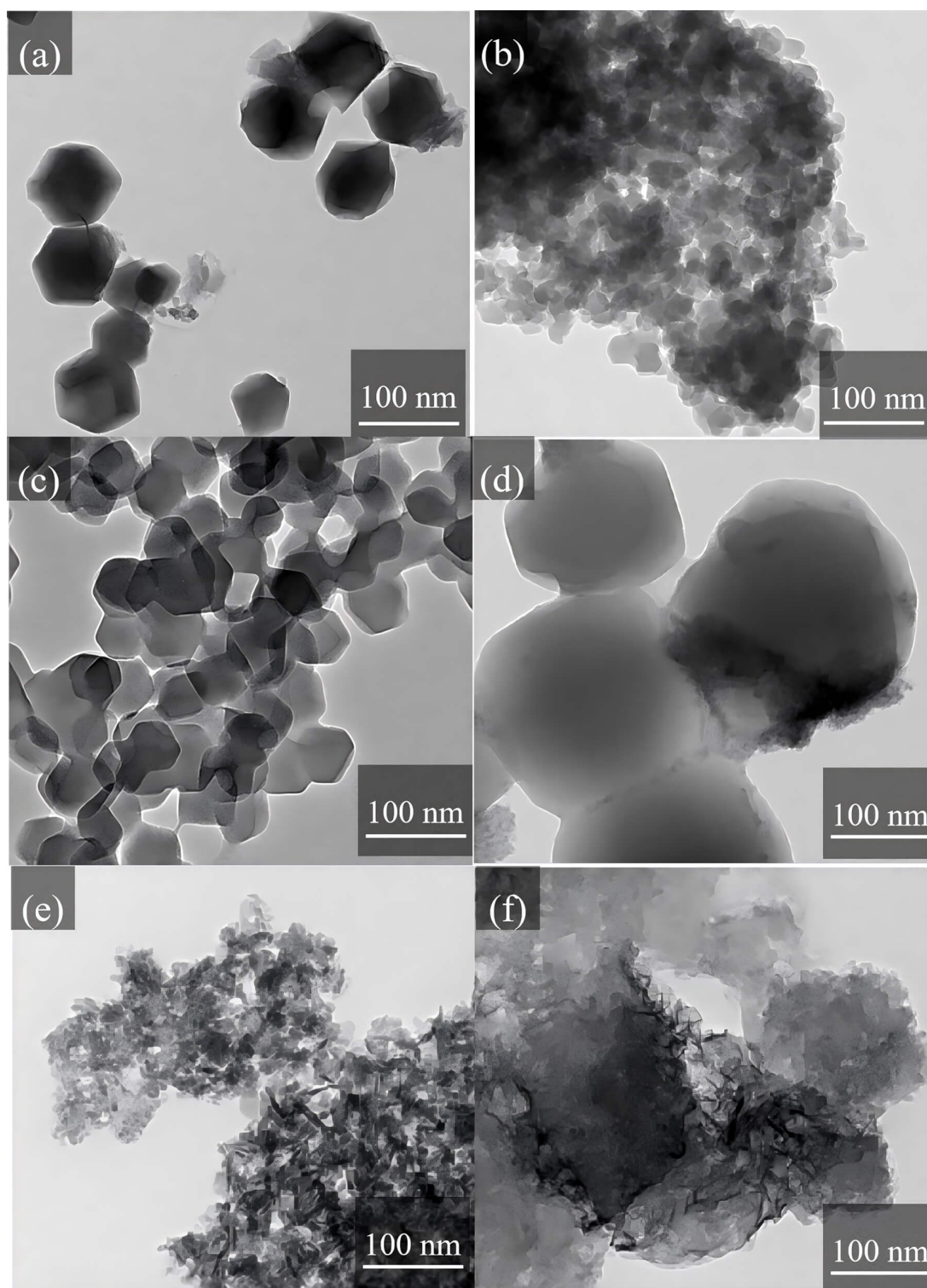


Fig. 2. Transmission electron microscopy (TEM) images before and after 0.5 mol/L NaCl solution containing 10 mg/L $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ treatment at 25 °C. (a) Zn-ZIF. (b) Co-ZIF. (c) Ni-ZIF. (d) Zn-ZIF ($\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$). (e) Co-ZIF ($\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$). (f) Ni-ZIF ($\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$). Scale bar = 100 nm.

From Fig. 5b, when Co-ZIF was added at a concentration of 50 mg/L, the E_{corr} of copper shifted positively and i_{corr} decreased markedly. From Fig. 5c, when the concentration of Ni-ZIF reached 50 mg/L, the E_{corr} of copper shifted pos-

itively and i_{corr} decreased, with a notable decline in i_{corr} . Generally speaking, compared with the blank group, if the shift in E_{corr} of the metal is less than 85 mV, the substance is considered a mixed-type inhibitor, i.e., it simultaneously

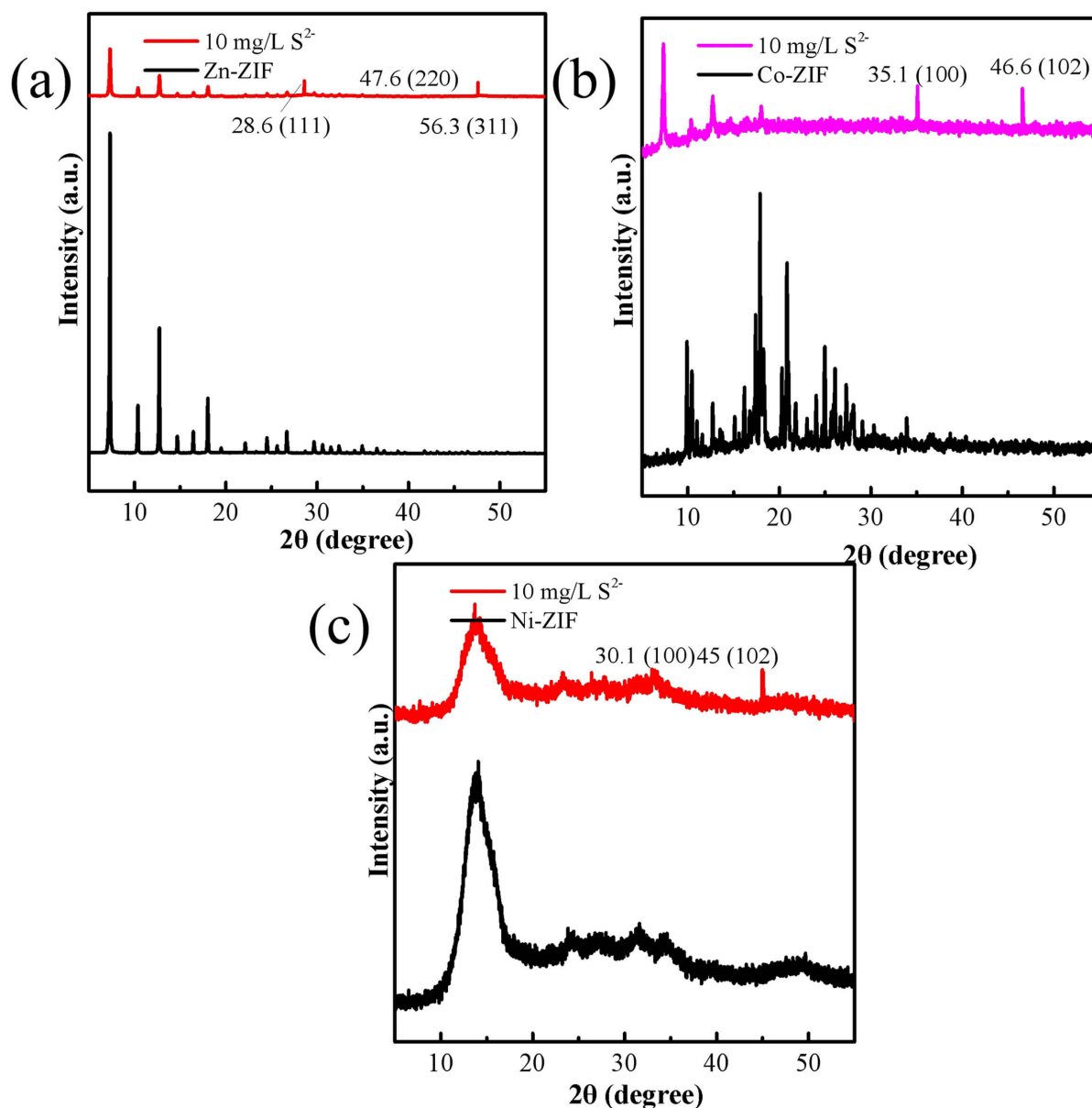


Fig. 3. X-ray diffraction (XRD) patterns of (a) Zn-ZIF; (b) Co-ZIF; (c) Ni-ZIF before and after $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ treatment at 25 °C.

inhibits both the anodic and cathodic reactions; if the potential shift exceeds 85 mV, the substance is regarded as a cathodic- or anodic-type inhibitor, which suppresses the metal corrosion reaction mainly by inhibiting either the cathodic or anodic reaction [43]. In Table 2, the addition of Zn-ZIF, Co-ZIF, and Ni-ZIF, the three ZIFs with different metal sources, resulted in corrosion potential shifts of less than 85 mV, indicating that all the studied ZIFs with different metal sources belong to the mixed-type inhibitor category. The data in Table 2 also show that at their optimal concentrations, the corrosion inhibition efficiencies of the three studied ZIFs with different metal sources for copper in NaCl solution were 89.31% (Ni-ZIF), 93.39% (Co-ZIF), and 90.06% (Zn-ZIF), respectively. These results indicate

that the corrosion inhibition capability of ZIFs varies depending on the metal source.

Secondly, the changes in the corrosion inhibition performance of ZIFs in the presence of S^{2-} were investigated. As can be seen from Fig. 5, with increasing S^{2-} concentration, the corrosion potential shifted gradually in the negative direction, and the corrosion current density increased progressively, indicating that the introduction of S^{2-} weakened the corrosion inhibition performance of ZIFs with different metal centers. The relevant parameters obtained from polarization curve fitting are listed in Table 2, from which it can be clearly observed that the corrosion inhibition efficiency exhibited a continuous downward trend with increasing S^{2-} concentration. When the S^{2-} concen-

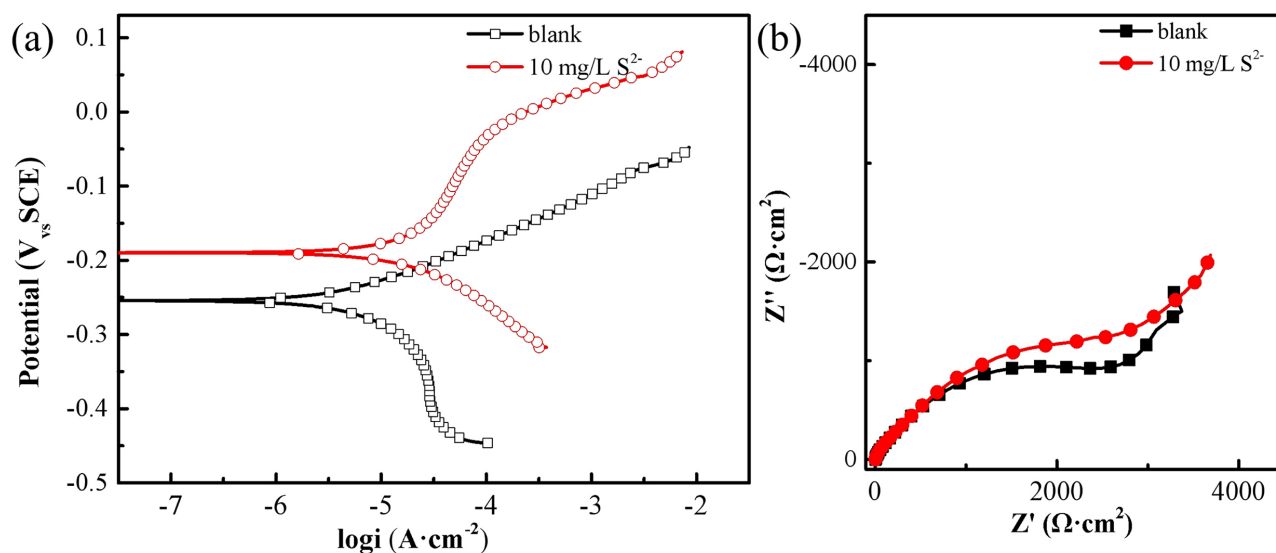


Fig. 4. Electrochemical measurements of copper in 0.5 mol/L NaCl solution with S²⁻ at 25 °C. (a) Polarization curves. (b) Nyquist plots.

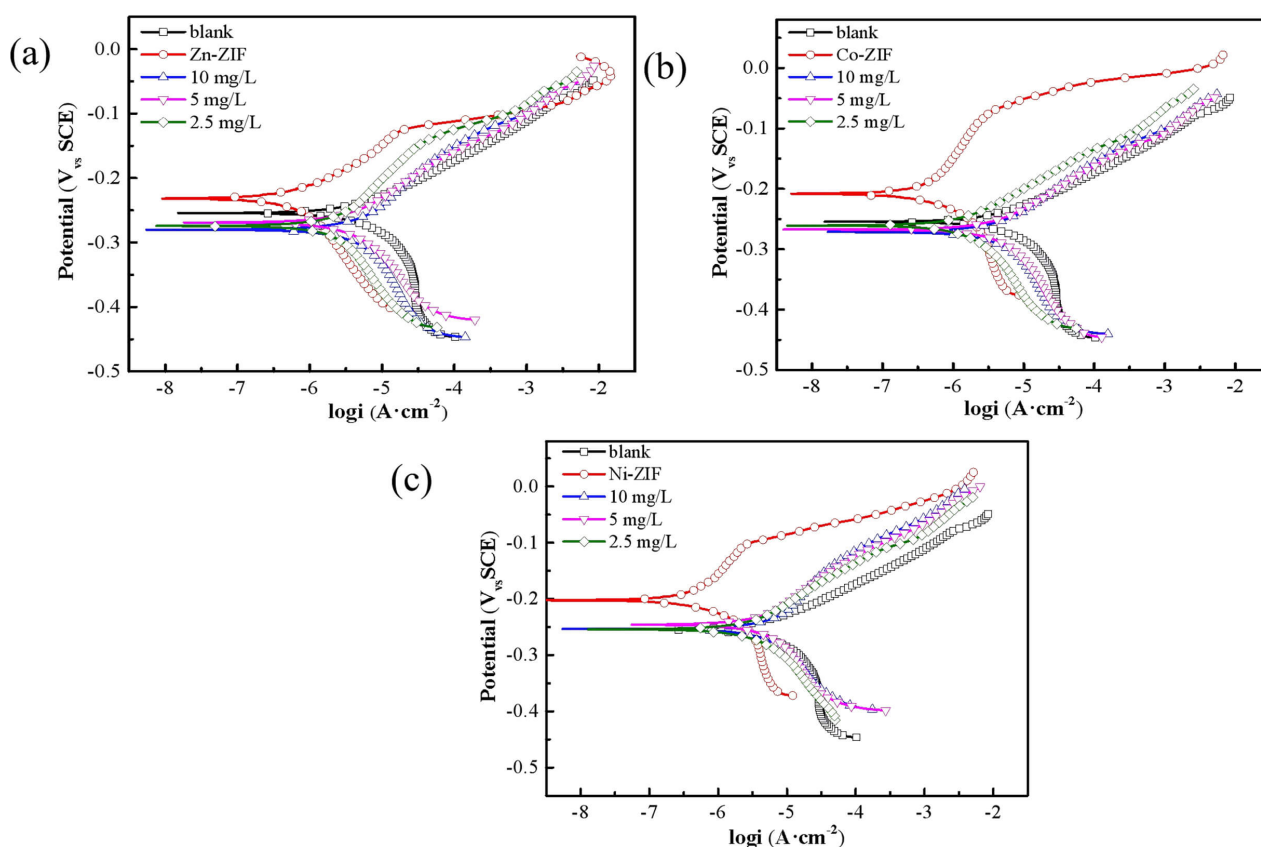


Fig. 5. Polarization curves of copper in 0.5 mol/L NaCl solution with different metal-center ZIFs and S²⁻ at 25 °C. (a) Zn-ZIF. (b) Co-ZIF. (c) Ni-ZIF.

tration reached 10 mg/L, the ZIFs with different metal centers lost their corrosion inhibition effectiveness almost completely. Through the analysis of the anodic and cathodic

Tafel slopes, a more detailed mechanistic interpretation of the polarization behavior was provided: intact ZIFs function as mixed-type inhibitors, capable of simultaneously

Table 2. Polarization curves fitted parameters of copper in 0.5 mol/L NaCl solution with S^{2-} and different metal-center ZIFs at 25 °C.

Samples	$C_{S^{2-}}$ (mg/L)	E_{corr} (mV)	b_a (mV/dec)	b_c (mV/dec)	i_{corr} ($A \cdot cm^{-2}$)	IE (η) (%)
	blank	−254	54	−112	4.75×10^{-6}	–
Zn-ZIF	0	−233	110	−54	4.72×10^{-7}	90.06
	2.5	−270	66	−80	3.64×10^{-6}	23.37
	5	−266	64	−83	3.77×10^{-6}	20.63
	10	−279	65	−81	3.99×10^{-6}	15.54
	0	−211	56	−101	3.14×10^{-7}	93.39
Co-ZIF	2.5	−256	63	−86	3.64×10^{-6}	23.37
	5	−258	63	−85	3.77×10^{-6}	20.63
	10	−263	62	−87	3.89×10^{-6}	18.11
	0	−202	123	−51	5.08×10^{-7}	89.31
Ni-ZIF	2.5	−252	69	−75	2.99×10^{-6}	37.05
	5	−253	68	−76	4.28×10^{-6}	9.89
	10	−255	75	−69	4.56×10^{-6}	4.00

suppressing anodic metal dissolution and cathodic oxygen reduction; whereas the increase in both anodic and cathodic current densities upon sulfide addition indicates that the breakdown of the ZIF framework weakened this dual inhibitory effect. From the perspective of adsorption characteristics, the protective film formed by intact ZIFs on the copper surface served as an effective barrier, while exposure to sulfide ions triggered a degradation process— S^{2-} seized the metal centers from the ZIF frameworks through competitive action, leading to structural collapse, which in turn caused the loss of the protective adsorption layer and ultimately resulted in a marked decline in corrosion inhibition efficiency.

$Na_2S \cdot 9H_2O$ was added to the 0.5 mol/L NaCl solution containing three different metal-center ZIFs to explore the effect of S^{2-} on the corrosion inhibition performance of these ZIFs. Impedance technology can provide further useful information for studying metal corrosion processes. Fig. 6 showed the impedance spectra of copper. As shown in Fig. 6, the impedance response curve of copper in the blank solution exhibits a concave semicircle at high frequencies and a straight line at low frequencies. The capacitive semicircle at high frequencies results from the double-layer capacitance and charge transfer resistance, which is typically interpreted as reflecting the charge transfer reaction; whereas the observed concave semicircle centered below the positive axis is attributed to metal interface inhomogeneities and other rough structures. The linear impedance curve at low frequencies corresponds to the Warburg impedance (W), reflecting the diffusion behavior of electroactive species, such as the transport of Cl^- ions/copper(II) chloride complexes or the diffusion of dissolved oxygen to the metal surface at the Cu–NaCl interface. Furthermore, in the presence of all three ZIFs, the Warburg impedance at low frequencies gradually disappears, and the curve diameter significantly increases compared to that of the blank solution, indicating that the ZIF

inhibitors exhibit strong protective effects. Therefore, the charge transfer process plays a pivotal role in copper corrosion. However, it can be seen from Nyquist Fig. 6a,c,e that the arc diameter of the capacitive resistance are gradually decreased with the increase S^{2-} concentration to 10 mg/L.

The Bode plots of the copper electrode under different conditions (Fig. 6b,d,f) exhibited distinct behavioral patterns compared to those in the blank solution. The relationship between $|Z|$ and $\log |f|$ in the Bode plots showed that upon addition of ZIFs, the impedance value increased significantly with a slope approaching -1 ; whereas in the presence of sulfide ions, the impedance value decreased progressively with rising ion concentration. The phase diagram of the blank copper electrode revealed two relaxation time constants, and two were also observed in the ZIFs-containing solution. Accordingly, two equivalent circuit models (Fig. 7a,b) were employed for fitting the EIS data. Furthermore, it is evident that ZIFs incorporation increased the phase angle toward 90° , indicating delayed corrosion progression.

And Fig. 7 was the equivalent circuit diagram used to fit the impedance data, where R_f represented the film resistance, R_s represented the solution resistance, R_{ct} represented the charge transfer resistance, and CPE represented the constant-phase element. The relevant data obtained by fitting are shown in Table 3. where CPE was represented by Eqn. 2 [44].

$$Z_{CPE} = \frac{1}{Y(jw)^n} \quad (2)$$

where Y was the CPE constant, w was the angular frequency, j was the imaginary root, n was the diffusion effect index, and when $n = 1$, CPE was pure capacitance. When $n = 0.5$, it was Warburg impedance; when $n = 0$, it was pure resistance; when $n = -1$, it was inductance. The closer the

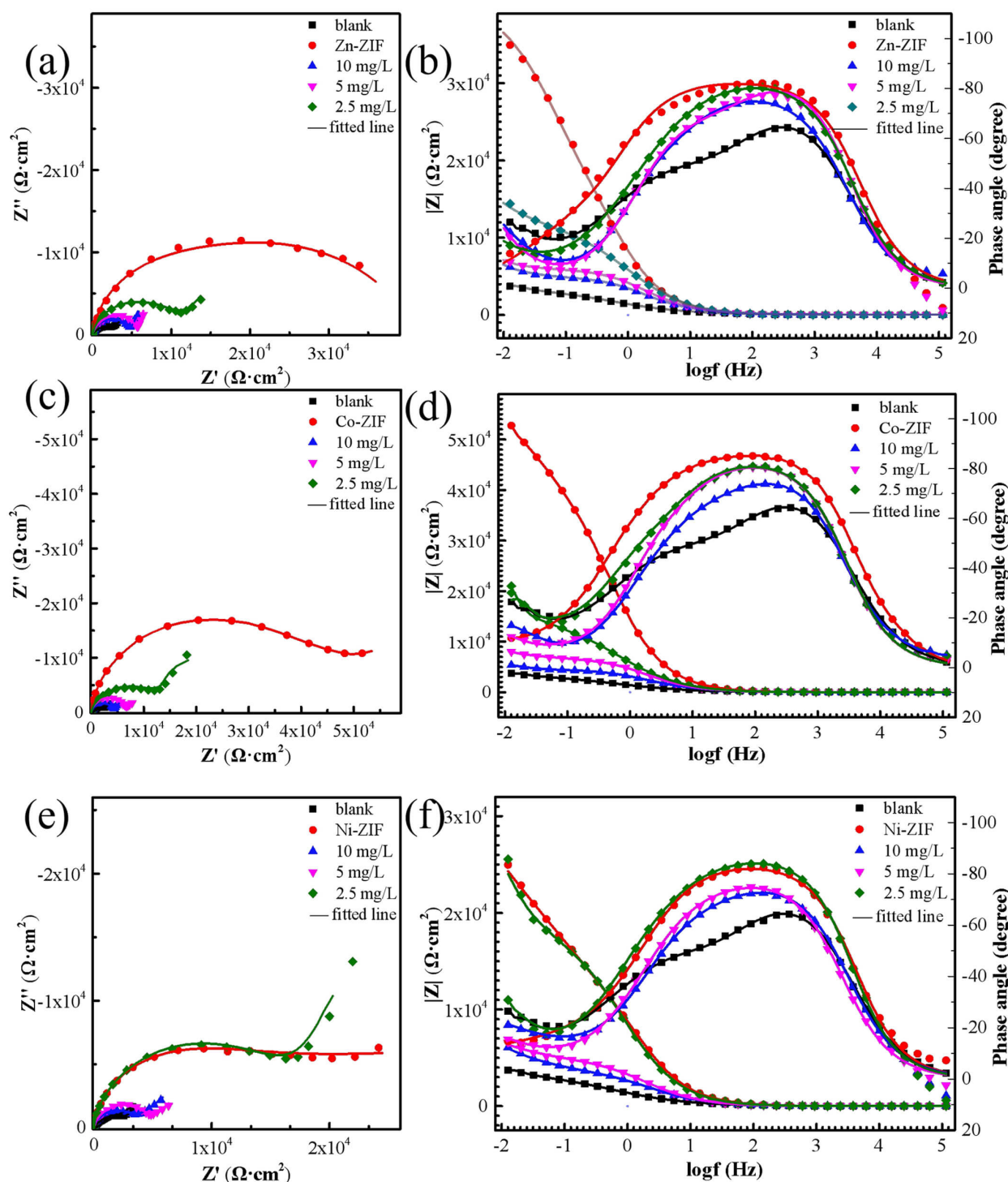


Fig. 6. Nyquist (a,c,e) and Bode plots (b,d,f) of copper in 0.5 mol/L NaCl solution with S^{2-} and different metal-center ZIFs at 25 °C. (a,b) Zn-ZIF. (c,d) Co-ZIF. (e,f) Ni-ZIF.

n value was to 1, the closer the CPE was to the pure capacitance. From Table 3, it was found that the R_f and R_{ct} increased significantly after the addition of three different metal center ion ZIFs compared to the blank conditions, indicating that these ZIFs covered the copper surface and formed a dense protective film on it. However, after increasing the S^{2-} concentration, R_f and R_{ct} continued to de-

cline. This indicates that the structure of the three different metal central ions ZIFs after S^{2-} treatment is changed, and they lose their ability to adsorb on the metal surface, resulting in a decrease in their corrosion inhibition effect.

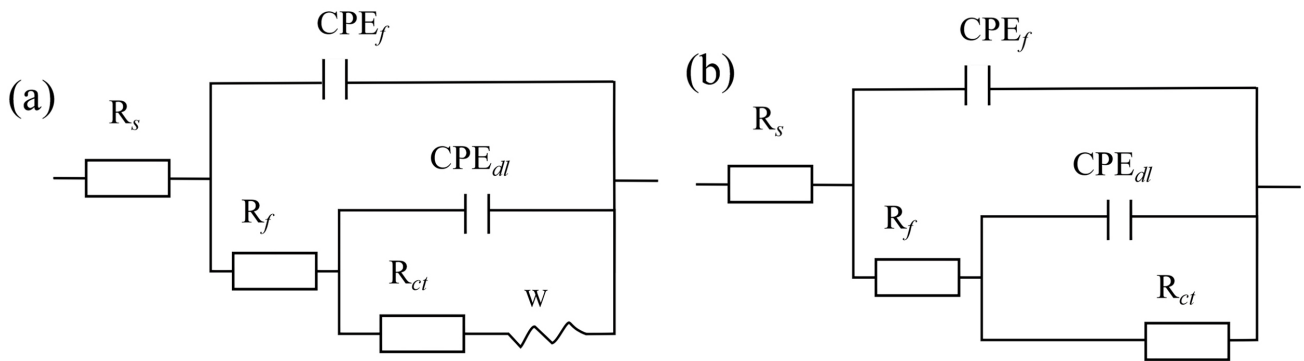


Fig. 7. Electrochemical equivalent circuits for fitting the impedance spectra (a) blank (b) ZIFs. CPE, Constant Phase Element. A CPE is used instead of a pure capacitor when the electrode surface is rough, heterogeneous, or when there is a distribution of relaxation times. CPE_f represents the non-ideal capacitance associated with a surface film or layer on the electrode. CPE_{dl} represents the non-ideal capacitance of the electrical double layer at the electrode/electrolyte interface. R_f , the film resistance; R_s , the solution resistance; R_{ct} the charge transfer resistance; W , the Warburg impedance.

Table 3. Fitted parameters of the EIS for copper in 0.5 mol/L NaCl solution with S^{2-} and different metal-center ZIFs at 25 °C.

Samples	$C_{S^{2-}}$ (mg/L)	R_s ($\Omega \cdot \text{cm}^2$)	CPE_f -T ($\text{S}^{n1} \cdot \Omega^{-1} \cdot \text{cm}^{-2}$)	n1	R_f ($\Omega \cdot \text{cm}^2$)	CPE_{dl} -T ($\text{S}^{n2} \cdot \Omega^{-1} \cdot \text{cm}^{-2}$)	n2	R_{ct} ($\Omega \cdot \text{cm}^2$)	Fitting E_{error}
blank	0	6	3.03×10^{-5}	0.87	290	1.54×10^{-4}	0.62	2928	0.043
	0	5	1.32×10^{-5}	0.96	9913	2.88×10^{-5}	0.66	14,443	0.037
	2.5	5	1.45×10^{-5}	0.93	3683	4.98×10^{-5}	0.72	9170	0.025
	5	5	1.13×10^{-5}	0.97	788	2.46×10^{-5}	0.74	5175	0.032
	10	6	2.44×10^{-5}	0.89	525	4.50×10^{-5}	0.76	4445	0.029
Zn-ZIF	0	6	8.75×10^{-6}	0.96	11,925	1.63×10^{-5}	0.79	20,867	0.040
	2.5	7	1.44×10^{-5}	0.94	4457	3.96×10^{-5}	0.71	8501	0.047
	5	6	1.69×10^{-5}	0.94	3553	3.38×10^{-5}	0.83	2786	0.028
	10	6	2.27×10^{-5}	0.89	1830	3.95×10^{-5}	0.78	2545	0.039
	0	6	1.06×10^{-5}	0.94	7551	6.97×10^{-5}	0.42	13,428	0.026
Co-ZIF	2.5	5	1.00×10^{-5}	0.98	4410	2.68×10^{-5}	0.55	10,483	0.018
	5	7	1.86×10^{-5}	0.92	2006	2.60×10^{-5}	0.58	2892	0.034
	10	6	2.54×10^{-5}	0.89	1040	3.41×10^{-5}	0.67	2344	0.041

EIS, electrochemical impedance spectroscopy.

3.1.2 SEM Results

The SEM diagram showed copper immersion in 0.5 mol/L NaCl containing S^{2-} and three different metal-center ZIFs corrosion in solution. As can be seen from Fig. 8a, the surface of copper soaked in blank solution was very rough and there are many corrosion products, which indicated that copper was severely corroded in neutral corrosion media. After adding three different metal center ion ZIFs (Fig. 8b–d), the copper surface was not only much smoother, but also has fewer corrosion traces, which indicated that the three different metal center ion ZIFs have a good inhibition effect on copper corrosion. However, the copper surface become rough in the presence of S^{2-} (Fig. 8e–g), which indicated that S^{2-} was not conducive to the adsorption of three different metal central ions ZIFs on the copper surface, resulting in a decrease in its corrosion inhibition properties.

4. Discussion

Based on the conclusions derived from comprehensive spectral characterization analysis, it is conclusively demonstrated that the crystal framework structures of all three ZIF materials involved exhibited significant degradation and dissociation to varying degrees. Dynamic potential polarization curve tests further revealed that in electrolyte simulation systems without sulfide ions (S^{2-}), the addition of these ZIF materials significantly reduced the corrosion current density on copper substrates, demonstrating effective corrosion inhibition. However, when S^{2-} was present in the system with increasing concentrations, the corrosion current density showed a progressive upward trend, clearly indicating that S^{2-} substantially diminishes or even reverses the corrosion-inhibiting efficacy of the ZIF materials. Additionally, equivalent circuit fitting analysis of electrochemical impedance spectra revealed that compared to the blank control group without any materials, the charge transfer resistance and surface protective film resistance in-

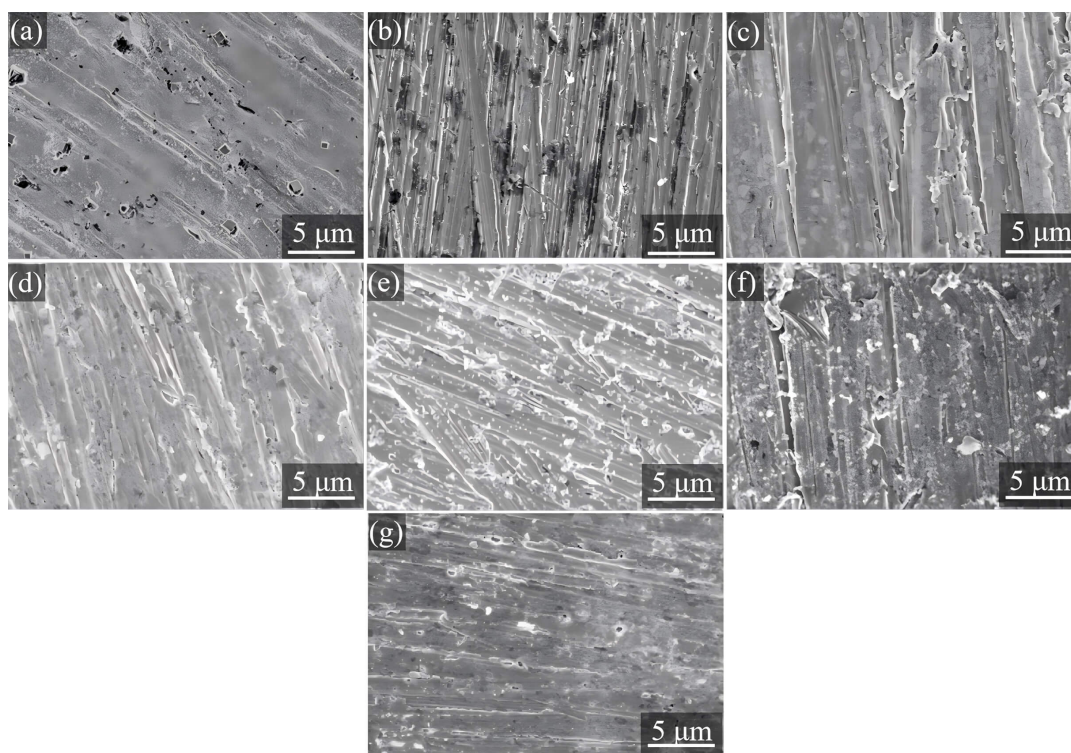


Fig. 8. SEM images of copper immersed in 0.5 mol/L NaCl solution containing different metal-center ZIFs and Na₂S·9H₂O for 24 h at 25 °C. (a) Blank. (b) Zn-ZIF. (c) Co-ZIF. (d) Ni-ZIF. (e) Zn-ZIF (Na₂S·9H₂O). (f) Co-ZIF (Na₂S·9H₂O). (g) Ni-ZIF (Na₂S·9H₂O). The 50 mg/L ions-ZIFs and 10 mg/L Na₂S·9H₂O. Scale bar = 5 μm.

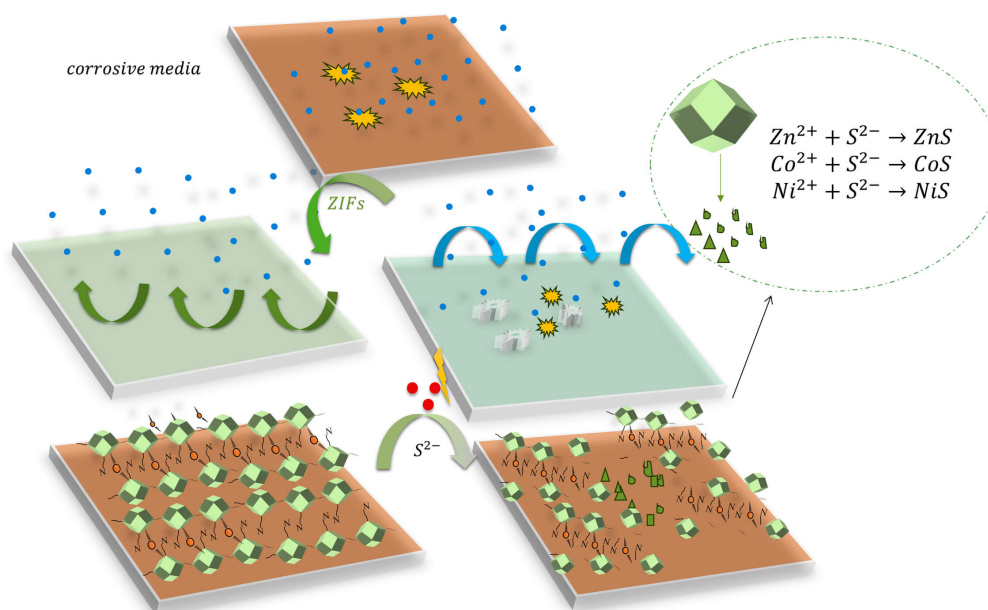


Fig. 9. Schematic diagram of the effect of S²⁻ on the corrosion inhibition behavior of different metal-center ZIFs.

creased markedly after ZIF material incorporation, while parameters characterizing the interfacial double-layer structure (e.g., constant-phase elements) significantly decreased. Given water molecules' high dielectric constant and their kinetic size being smaller than the average pore size of the studied ZIF materials, the substantial reduction in interfa-

cial capacitance primarily results from ZIF materials effectively displacing and replacing the original water layer on the copper interface upon adsorption. Conversely, the charge transfer resistance and surface film resistance exhibited a clear decline in presence of S²⁻. These findings collectively confirm that S²⁻ severely interferes with and

compromises the effective adsorption and stabilizing coating capabilities of ZIF materials on copper surfaces.

As illustrated in Fig. 9, by integrating various spectral characterization techniques with electrochemical test results, this study provides a comprehensive elucidation of the corrosion inhibition mechanism and failure process of ZIF materials against copper in the presence of S^{2-} . The underlying mechanism involves two key steps: First, in an absence of S^{2-} , metal center ions (e.g., Zn^{2+} , Co^{2+} , Ni^{2+}) within ZIF materials interact strongly with nitrogen-containing heterocyclic organic ligands (such as 2-methylimidazole) through robust metal-ligand coordination, forming a dense protective adsorption layer on the copper surface that effectively prevents direct contact between corrosive media and the substrate. However, when S^{2-} is present, it rapidly interacts with metal center ions via ion competition adsorption, forming highly soluble metal sulfide precipitates with low solubility. This process vigorously dislodges imidazole-based ligands from metal centers, leading to irreversible degradation of the ZIF molecular framework and rendering it incapable of forming a complete protective layer on copper surfaces, thereby completely compromising its corrosion protection efficacy [29,45].

Limitations

This study has several limitations that should be acknowledged. First, the characterization techniques employed to probe the structural evolution and failure mechanism of the MOFs were limited. In particular, X-ray photoelectron spectroscopy (XPS) and atomic force microscopy (AFM) were not utilized; these techniques could provide direct chemical state information on the metal-sulfide interactions and nanoscale topographical evidence of protective film degradation, respectively. Their absence limits the depth of mechanistic interpretation at the interface. Second, the study focused exclusively on three ZIF-type MOFs and a single aggressive ion species (S^{2-}) under static immersion conditions, which may not fully capture the complexity of real-world service environments involving multiple coexisting ions and hydrodynamic factors. Future work will incorporate XPS and AFM analyses to strengthen the mechanistic understanding and will extend the investigation to a broader range of MOF structures and multi-ion systems under flow conditions to enhance the generalizability of the findings.

5. Conclusion

In conclusion, this paper employed electrochemical methods to monitor the alterations in the corrosion inhibition performance of various zeolitic imidazolate frameworks (ZIFs) within an S^{2-} -containing environment. Additionally, it investigated the influence mechanisms of relevant factors on the corrosion inhibition behavior through characterization analysis. The research indicates that in the

presence of S^{2-} ions, competition occurs, causing the seizing of metal ions from the structures of three ZIFs with distinct metal centers, namely Zn-ZIF, Co-ZIF, and Ni-ZIF. This leads to the collapse of their frameworks, and subsequently results in a significant decrease in the corrosion inhibition performance of these ZIFs. This finding holds significant importance for a more in-depth understanding of the stability and reliability of ZIFs in practical applications, emphasizing the necessity of considering the risks posed by external environmental factors when selecting ZIF-based corrosion inhibitors.

Availability of Data and Materials

The datasets used or analyzed during the current study are available from the corresponding author on reasonable request.

Author Contributions

JC: Writing-review & editing, Writing-original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation. JG and YG: Writing-review & editing, Supervision, Methodology, Funding acquisition, Conceptualization. All authors read and approved the final manuscript. All authors have participated sufficiently in the work and agreed to be accountable for all aspects of the work.

Ethics Approval and Consent to Participate

Not applicable.

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Conflicts of Interest

The authors declare no conflicts of interest.

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