

## Research Article

# Fatty Amidine as Copper Corrosion Inhibitor

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The development of green and sustainable corrosion inhibitors for copper in a corrosive marine environment is highly desired. Herein, we studied the fatty acid-based amidine as the new type of renewable corrosion inhibitor. Stearamidine salt was used as a model inhibitor, and it was synthesized through stearonitrile intermediate with an excellent isolated yield of 88%. We used electrochemical (potentiodynamic polarization) and morphological (scanning electron microscopy) measurements to assess the corrosion inhibition efficiency of stearamidine in 3.0 wt.% NaCl at 300 K. We show that, in such a condition, the optimum inhibition efficiency of 96% was achieved using only 0.2 mM stearamidine. The results suggested the fatty amidine is a promising corrosion inhibitor for copper that is suitable in the saltwater ecosystem. The thermodynamic parameters of the interaction between the stearamidine and the copper surface were determined, and the result suggests that the adsorption process occurred accordingly with the Langmuir adsorption isotherm and involved both physisorption and chemisorption.

## 1. Introduction

Materials based on copper and copper alloys are commonly used for various types of industrial equipment. These materials offer a particular advantage in a combination of thermal and/or electrical conductivity, mechanical workability, and it is relatively inert [1]. They are typically applied as heat exchangers, power plant condensers, water-cooling systems, desalination plants, and shipboard condensers. Copper and copper alloys are prone to corrosion in various environments, especially in marine environments. The corrosion on copper and its alloys takes place at a higher rate in the presence of high concentration of salt ions, which will culminate in the structural rupture and create enormous economic losses. The addition of corrosion inhibitors on the metal surfaces is one of the most effective, economical, and conventional approaches to prevent this detrimental process [1–3].

The aim to add corrosion inhibitor on a metal surface, usually in low concentration, is to effectively minimize the corrosion rate of the substrate in a specific environment,

such as the highly corrosive marine ecosystem. Some organic molecules have been reported as potential compounds that can effectively inhibit corrosion for various metals and metal alloys, including copper and its alloys [4]. These compounds show high inhibiting properties due to their adsorption affinity with copper surfaces through intermolecular interactions [5]. It has been known that the chemical structure, the adsorption mode, and the surface charge of metals, including the type of electrolyte solution, are the key points governing the interactions of molecules on the surfaces of metals [6]. It is not surprising that the reported corrosion inhibitor compounds are derived from azoles, imidazoles, triazoles, indazole, oxazole, thiadiazole, and quinoline framework [7–13]. These inhibitors contain heteroatom centres such as sulphur, oxygen, and nitrogen acting as their major adsorption sites. Due to the inevitable trend toward environmentally friendly and sustainable processes, current research is focused on the development of green corrosion inhibitors [14–18]. One type of compound that shows promising potential as a corrosion inhibitor of copper is the

derivatization of oleochemicals that can be obtained from a renewable source such as the palm oil industry. The presence of long alkyl chains can help to form a protective layer and the terminal moiety that can be easily manipulated to tune the binding properties on metal surfaces.

Fatty acid derivatives are inexpensive, abundant, and ideal candidates as precursor materials for producing sustainable and green chemicals [19, 20]. There are numerous reports regarding the corrosion inhibitors for metal surfaces based on the fatty acid derivatives, including acyclic and heterocyclic compounds [21–31]. Interestingly, there are no reports of fatty amidine as a corrosion inhibitor. Amidine is a derivative of oxoacid with the general structure of the  $R_nC(=NR)NR_2$ , wherein the hydroxyl group and oxo group were substituted by an amino group and imine group, respectively [32].

In the present study, we use stearamidine hydrochloride salt as a model compound to evaluate the potential corrosion inhibition of fatty amidine on a copper surface. To assess the corrosion inhibition efficiency of stearamidine in 3.0 wt.% NaCl, we used electrochemical and morphological analysis. The thermodynamic parameters were also calculated and discussed, and thus the nature of their interaction is also elucidated.

## 2. Methods

**2.1. Materials.** All chemical reagents were brought from commercial sources without further purification. Characterizations were conducted by FTIR (IR Prestige-21) and GC (GC 2010) from Shimadzu. NMR spectra were measured using an ECA 500 JEOL. The chemical shifts ( $\delta$ ) of  $^1H$  NMR were showed in the unit of ppm, and the value of Hertz (Hz) is used for the coupling constants ( $J$ ). The referenced chemical shift of deuterated solvents is  $CD_3OD$  (3.31 ppm) and  $CDCl_3$  (7.26 ppm). The electrochemistry measurements were conducted on a CH750 potentiostat from CH Instrument workstation. Silver (Ag/AgCl), platinum (Pt), and copper (Cu) were used as the reference, counter, and working electrodes, respectively.

### 2.2. Procedure

**2.2.1. Stearonitrile.** Stearic acid (60.2 g, 0.211 mol) was put into a two-neck flask and was melted at  $80^\circ C$  under nitrogen atmosphere. Urea (30 g, 0.499 mol) was put in gradually, and then the flask is heated with vigorous stirring at  $300^\circ C$  for 6 h. The reaction progress was monitored by TLC analysis. After the reaction is complete, the subsequent mixture was filtered through Celite. The filtrates were separated by recrystallization. Further purification was done with column chromatography by using a solvent mixture of *n*-hexane and ethyl acetate with 6:1 ratio. The final compound was obtained as off-white solid, and the yield is 92%. Melting point was  $40\text{--}41^\circ C$  (stearonitrile standard:  $41\text{--}41.5^\circ C$ ). FTIR (KBr)  $\tilde{\nu}$  was  $2249\text{ cm}^{-1}$  (stretch  $C\equiv N$ ),  $2920$ , and  $2851\text{ cm}^{-1}$  (stretch C-C and C-H, respectively).  $^1H$  NMR (500 MHz,  $CHCl_3$ )  $\delta$  was 2.33 (t, 2H); 1.63 (m, 2H); 1.39 (m, 2H); 1.25 (s, 26H); and 0.95 (t, 3H). Purity was examined by GC: 98%.

**2.3. Synthesis of Stearamidine Hydrochloride.** A total of 3 g stearonitrile compound was dissolved in 33 mL of methanol-diethyl ether mixture. Dried hydrochloric acid gas (HCl) was passed through the solution for two hours at  $0^\circ C$  and then cooled to  $-20^\circ C$  for 96 hours. The solvent was evaporated to produce imide hydrochloride and then washed with cold diethyl ether solution. Imide hydrochloride was treated with 100 mL of ammonia in methanol ( $NH_3/MeOH$  7N, 5 eq.). The obtained filtrate was then separated by filtration and evaporation to obtain stearamidine hydrochloride salt as off-white solid. The yield was 88.2%. FTIR (KBr)  $\tilde{\nu}$ , in  $cm^{-1}$ , was 3215 (stretch N-H), 3000 to 2800 (stretch C-C and C-H), 1648 ( $C=N$  symmetric vibration), 1415 (stretch  $C=N$ ), and 1092 (stretch C-N), and  $^1H$  NMR (500 MHz,  $CD_3OD$ )  $J = 2.46$  (t, 2H), 1.70 (m, 2H); 1.35 (m, 28H); and 0.86 (t, 3H).

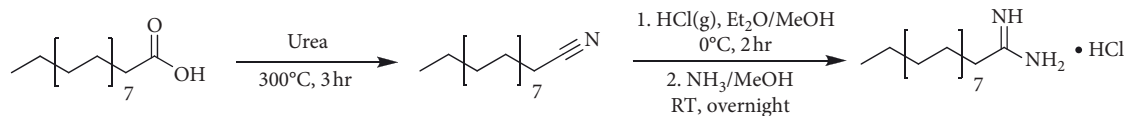
**2.4. Corrosion Inhibition Measurement.** The electrochemical scan was conducted from cathode to anode with potential in the range of  $-400$  to  $+400$  mV vs. saturated calomel electrode (SCE) at  $0.5\text{ mV s}^{-1}$  sweep rate at three different temperatures (300, 313, and 333 K) by using various concentrations of stearamidine. The measured current density was used for the calculation of corrosion inhibition efficiency ( $\eta$ ) by using the formula as follows:

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

where the efficiency of inhibition is  $\eta(\%)$ , the current density in blank solution is  $i_{\text{corr}}$ , while the current density in a solution with the presence of stearamidine is  $i_{\text{corr}}^0$ . The sample with optimum inhibition efficiency was then analyzed for its surface morphology by using SEM, and its surface micrograph was compared with the corresponding blank sample.

## 3. Results and Discussion

**3.1. Synthesis and Characterization of Stearamidine Hydrochloride.** We used stearamidine as a model compound to evaluate the potential application of fatty amidine as a green copper corrosion inhibitor. Stearamidine salt was synthesized from free fatty acid in two steps of reaction. Scheme 1 shows the reaction route for the synthesis of stearamidine. Based on Scheme 1, firstly, stearic acid was reacted with urea at  $300^\circ C$  for 2 hours to afford stearonitrile as off-white solid [33]. The GC analysis of the synthesized stearonitrile revealed that the purity of the product was very high, around 98%. After the nitrile was obtained, the preparation of stearamidine salt was conducted via Pinner synthesis developed by Baker et al. [34]. The treatment of nitriles with gaseous HCl in a mixture of anhydrous ether and methanol produced an imino ester. Then, the salt was reacted with ammonia to obtain amidine hydrochloride. The completion of reaction was indicated by the presence of a new spot at the  $R_f$  value about 0.41 and the disappearance of the spot corresponding to stearonitrile ( $R_f$  of 0.83) (see Figure 1(a)). The product after purification was isolated as a white solid with a good reaction yield of 88%.



SCHEME 1: Synthesis route of stearamidinium.

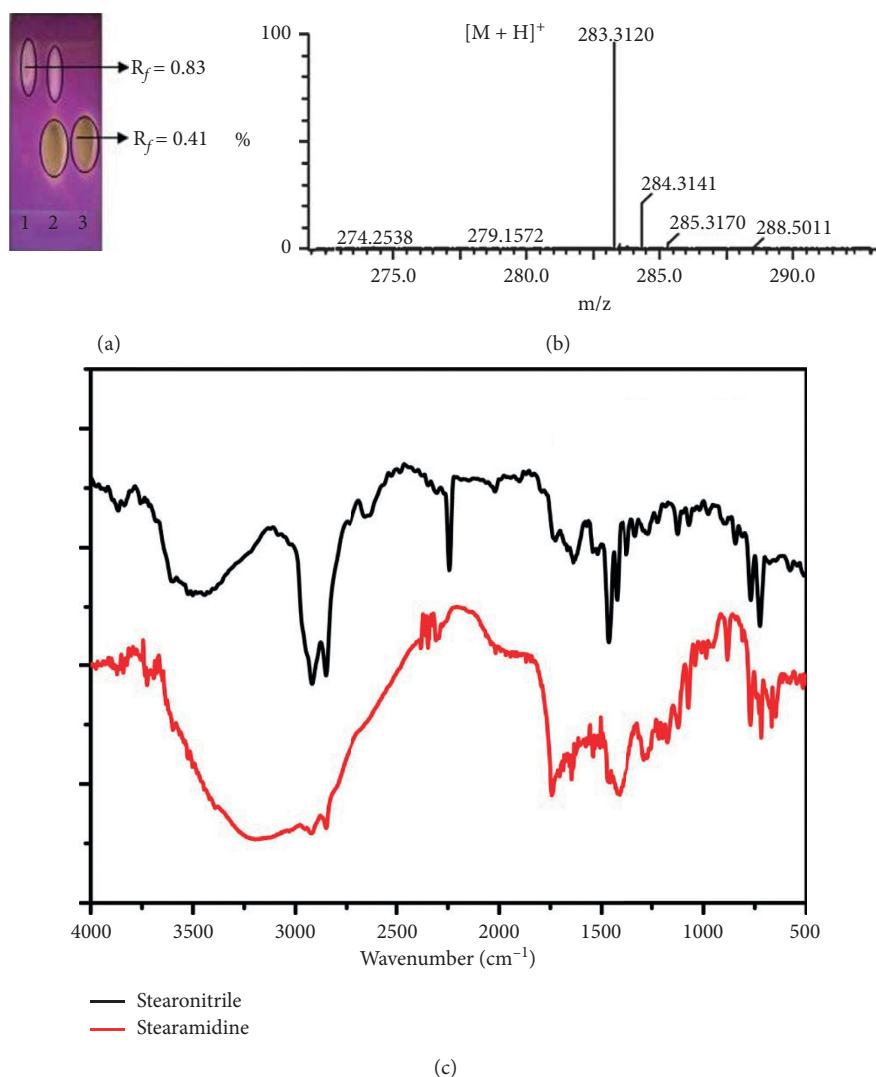


FIGURE 1: (a) TLC of reaction crude (1 = precursor, 2 = cospot, and 3 = reaction crude). (b) ESI MS of stearamidinium. (c) FTIR spectra of stearonitrile (black) and stearamidinium (red).

In Figure 1(b), the ESI mass spectrum of the isolated product confirms that the isolated compound is stearamidinium with one major peak at 283.312  $m/z$ , which is in good agreement with the calculated MW of stearamidinium  $[M + H]^+$ . FTIR spectrum of synthesized stearamidinium is presented in Figure 1(c). The broad peaks in the region ranging from 3200 to 3500  $\text{cm}^{-1}$  belong to the N–H stretching vibrations, and these bands are not detected in the FTIR spectrum of stearonitrile. On the other hand, the lacking of the CN stretch band in the region of 2210–2260  $\text{cm}^{-1}$  indicates that the conversion of nitrile is completed. Moreover, the bands corresponding to the presence of  $-\text{CH}_2-$  groups at

2870 and 2956  $\text{cm}^{-1}$  are also detected. Furthermore, to verify that stearamidinium was obtained, we investigated the NMR spectra of the product. The proton NMR analysis confirms that the product is stearamidinium. As can be seen from Figure 2, four distinct peaks are observed in the NMR spectra and correspond to four different types of hydrogen that are present in stearamidinium, with  $J = 2.46$  (t, 2H); 1.70 (m, 2H); 1.35 (m, 28H); and 0.86 (t, 3H), respectively.

The polarization curves of samples were measured in 3.0 wt.% NaCl solution at 300 K with various concentrations of stearamidinium (Figure 3). The electrochemical parameters were then calculated from the corresponding polarisation

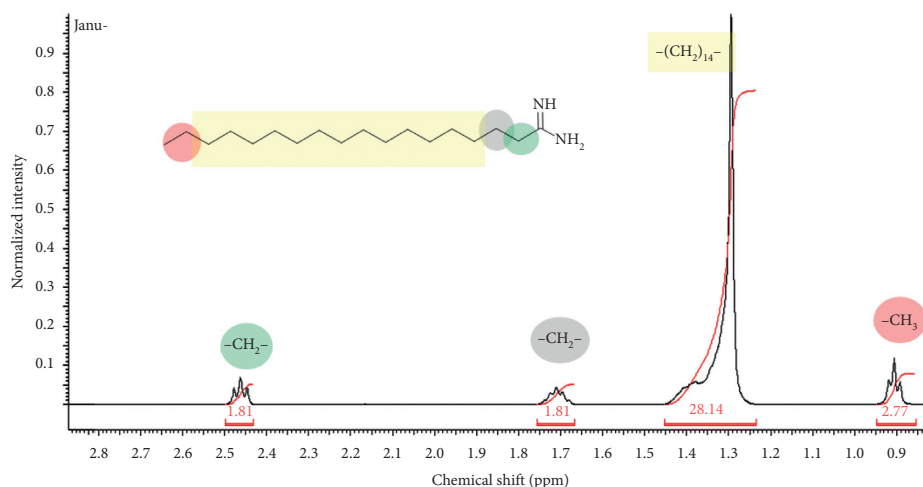


FIGURE 2: Proton NMR spectra of the product. Inhibition performance: potentiodynamic polarisation and SEM imaging.

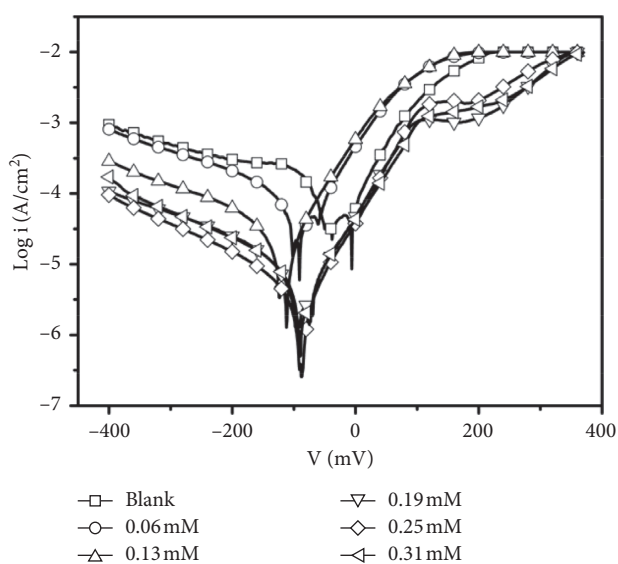


FIGURE 3: Potentiodynamic polarization curves of Cu in 3.0 wt.% NaCl solution at 300 K with a variety of concentrations of stearamidide.

curves (Table 1). In the blank solution, it was observed that the anodic current density consistently increased with potential (see Figure 3) and is in good agreement with previous reports [8, 12]. This result represents the dissolution of the Cu electrode to generate soluble species, i.e.,  $\text{CuCl}_2$ . The current density then decreases after reaching a maximum point. This trend corresponds to the formation and arrangement of the  $\text{CuCl}$  thin film. Dissolution of this thin deposition to produce  $\text{Cu(II)}$  ions further increases the current density again. In contrast, the addition of stearamidide leads to a substantial drop in the corrosion rate. The presence of stearamidide shifts and decrease in cathodic and anodic current densities hence inhibits the corrosion rate in comparison with the blank solution at the corresponding individual potential (Figure 3).

The anodic and cathodic reaction mechanisms were not changed by the addition of stearamidide as the inhibitor, as

each curve in Figure 3 was found to be parallel to each other. This result indicated that stearamidide adsorbed on the copper surfaces act as a protective layer providing a high degree of protection from the corrosion process [3]. Moreover, the change of  $E_{\text{corr}}$  value in the presence of stearamidide is less than 85 mV in comparison with the blank solution. This result confirms that stearamidide is a mixed-type corrosion inhibitor [35]. With the increasing of stearamidide concentration, we found a drop in the value of corrosion current density. At the same time, there was an increase of the inhibition efficiency (EI) and reached a maximum value of 96% when using 0.2 mM stearamidide in 3.0 wt.% NaCl solution at 300 K (Table 1). The increase of EI with the concentration is expected because with an increase in the number of stearamidide molecules, more corrosion sites can be blocked. Amidine moieties possess a donor electron group from the lone electron pair of the nitrogen atom. This lone pair can be donated to the positively charged copper surfaces forming a strong coordination bond that ensures excellent inhibition efficiency.

To elucidate the influence of temperature on the corrosion inhibition properties, calculated inhibition efficiency (EI), and corrosion rate (CR) of the stearamidide, the electrochemical measurement was conducted by varying the concentration of stearamidide at applied temperatures, in the range of 300 to 333 K (Figure 4). The result indicated that temperature has a damaging impact on the ability of stearamidide to inhibit the corrosion process. As the temperature increases, the EI value decreases significantly while the corrosion rate increases. This phenomenon is mainly due to the properties of organic molecules, in which the adsorption capacity is weakened at high temperatures. At high temperatures, adsorption and desorption rates from the metal surface are relatively high. Besides, at high temperatures, the organic-based inhibitor compounds are possible to be decomposed [6]. We also investigated the effect of stearamidide on the surface morphology of copper. The SEM micrographs of copper with and without the addition of stearamidide are presented in Figure 5. Clearly, Figure 5 shows that the addition of stearamidide as corrosion

TABLE 1: Electrochemical parameters of Cu in 3.0 wt.% NaCl solution at 300 K with various concentrations of stearamidine.

Stearamidine (mM)	Tafel slopes		E <sub>corr</sub> (mV)	I <sub>corr</sub> (x10 <sup>-6</sup> A/cm <sup>2</sup> )	Θ	CR (mm cm <sup>-2</sup> h <sup>-1</sup> )	EI (%)
	Cathode	Anode					
Blank	$y = -0.004x - 3.960$	$y = 0.015x - 4.577$	32.47	81	—	0.00052	0.00
0.06	$y = -0.004x - 4.464$	$y = 0.013x - 3.766$	-41.10	50	0.383	0.00032	38.27
0.13	$y = -0.005x - 5.079$	$y = 0.013x - 3.644$	-79.72	21	0.741	0.00014	74.07
0.19	$y = -0.005x - 5.535$	$y = 0.013x - 4.759$	-43.11	4.8	0.941	0.00003	94.07
0.25	$y = -0.005x - 5.778$	$y = 0.013x - 4.837$	-52.28	3.0	0.963	0.00002	96.30
0.31	$y = -0.005x - 5.508$	$y = 0.012x - 4.741$	-45.12	5.2	0.936	0.00003	93.58

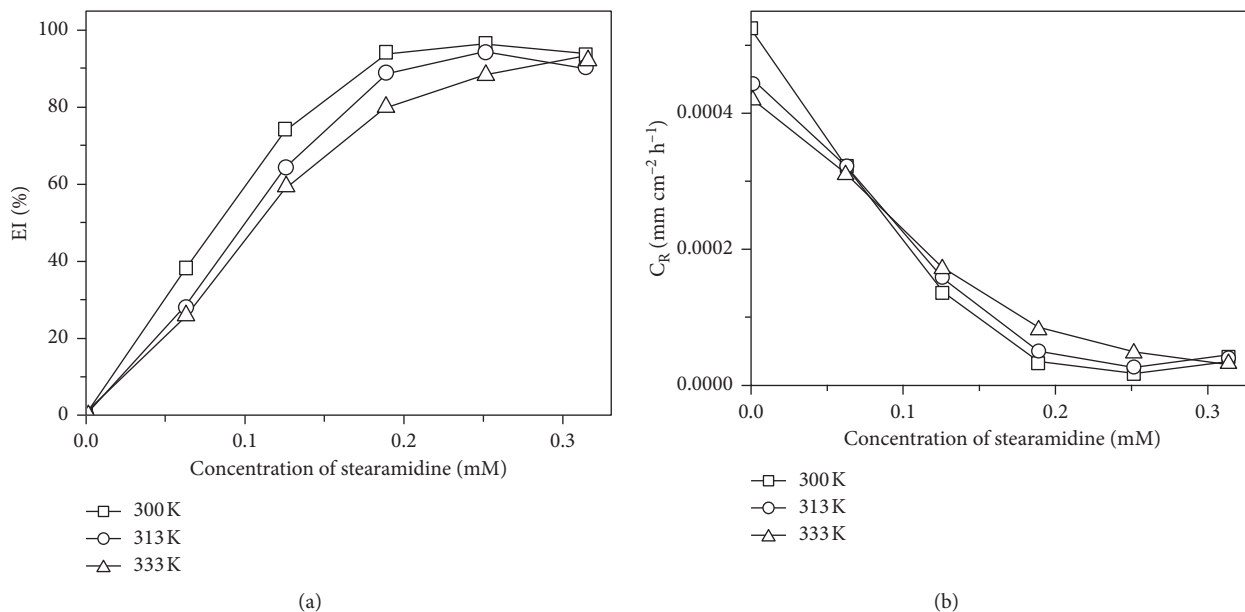


FIGURE 4: Inhibition efficiency (a) and corrosion rate (b) of copper as a function of the concentration of stearamidine in 3.0 wt.% NaCl solution at 300–333 K.

inhibitor enhances the metal resistance to corrosion. Figure 5(a) shows that the oxidation reaction occurs at the surfaces of copper, leading to the rupture of metal structure initiated by microporous formations on its surfaces. On the other hand, by addition of stearamidine, the surface of copper is well-protected from oxidation (Figure 5(b)).

**3.2. Isotherms of Adsorption.** It is essential to evaluate the adsorption isotherm to reveal the inhibition mechanism of stearamidine on copper surfaces. Based on data fitting with well-known isotherm models (i.e., Langmuir, Frumkin, Freundlich, and Temkin) [36], it was found that the inhibition of stearamidine followed the Langmuir-type adsorption. The equation of Langmuir adsorption is as follows:

$$\frac{[C]}{\theta} = \frac{1}{K_{\text{ads}}} + [C], \quad (2)$$

where the surface coverage is  $\theta$ , the adsorptive equilibrium constant is  $K_{\text{ads}}$ , and the concentration of stearamidine is  $[C]$ .  $[C]/\theta$  as a function of  $[C]$  at three different temperatures

(300, 313, and 333 K) is shown in Figure 6. The  $K_{\text{ads}}$  value of stearamidine for each temperature was estimated from the linearly fitted curve reported in Figure 6. The estimated  $K_{\text{ads}}$  at different temperatures are summarized in Table 2.

To determine the free energy parameter of the stearamidine adsorption on the copper surface ( $\Delta G_{\text{ads}}^0$ ), we used the following relation:

$$\Delta G_{\text{ads}}^0 = -RT \ln(55.5 K_{\text{ads}}), \quad (3)$$

where  $T$  is the absolute temperature (K) and  $R$  is the universal gas constant, and the concentration of water used in the solution is 55.5. Table 2 provided the value of calculated  $\Delta G_{\text{ads}}^0$  that is in the range of  $-31.61$  to  $-35.29$  kJ mol<sup>-1</sup>. These negative values indicate that the adsorption phenomenon, where a stable molecular layer of stearamidine on the copper surface is formed, is a spontaneous process. It is reported that the type of adsorption is regarded as physisorption if the  $\Delta G_{\text{ads}}^0$  values are below  $-20$  kJ mol<sup>-1</sup>. At this level, the inhibition activity is mainly dominated by Coulombic interactions. On the other hand, if the  $\Delta G_{\text{ads}}^0$  values are higher than  $40$  kJ mol<sup>-1</sup>, the adsorptions are predominately by chemisorption process [35]. Therefore, by evaluating  $\Delta G_{\text{ads}}^0$ ,



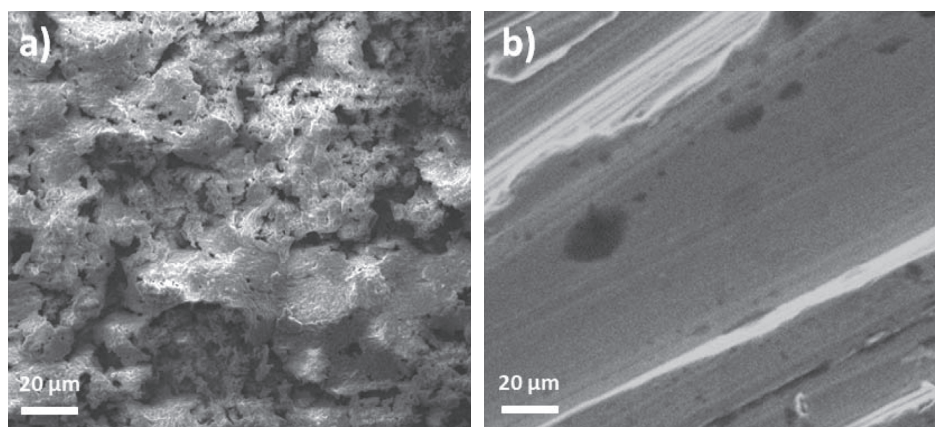


FIGURE 5: SEM micrographs of copper surfaces (250x magnitude). (a) Copper surface without corrosion inhibitor. (b) Copper surface with corrosion inhibitor at optimum condition (0.2 mM stearamidine, 300 K).

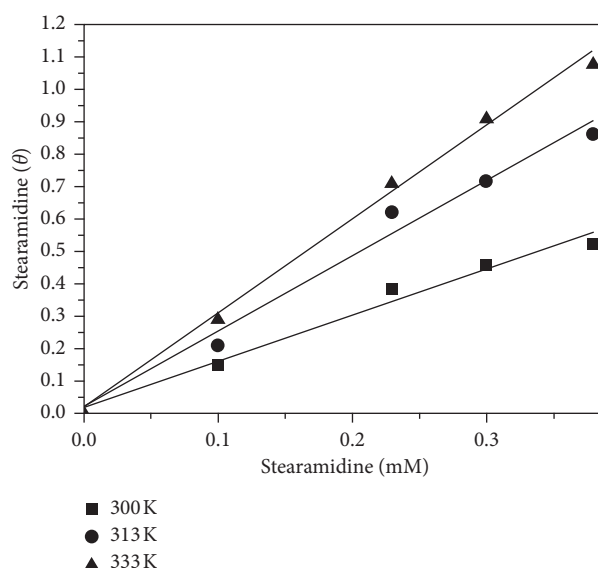


FIGURE 6: Langmuir-type adsorption isotherms of stearamidine in 1 wt.% NaCl solution at 300–333 K.

TABLE 2: Calculated thermodynamic parameters of the interaction between stearamidine and copper surface in 3.0 wt.% NaCl solution at 300–333 K.

Compound	$T$ (K)	$R$	$K_{\text{ads}}$ ( $\text{L mol}^{-1}$ )	$\Delta G^\circ$ ( $\text{kJ mol}^{-1}$ )	$\Delta H^\circ$ ( $\text{kJ mol}^{-1}$ )	$\Delta S^\circ$ ( $\text{J mol}^{-1}$ )
Stearamidine	300	0.999	5777.01	-31.61	34.77	-11.86
	313	0.992	4037.14	-32.05	36.28	-11.36
	333	0.998	6222.78	-35.29	38.59	-10.68

the stearamidine can be classified as a mixed-type inhibitor in which its adsorption on copper surfaces involves physisorption and chemisorption processes.

#### 4. Conclusions

Fatty amidine has been evaluated as a potential corrosion inhibitor for copper using stearamidine as a model molecule. Stearamidine hydrochloride is successfully synthesized from stearic acid (yield 88%). Based on SEM images and potentiodynamic measurements, stearamidine shows

excellent potential as a corrosion inhibitor for copper in 3.0 wt.% NaCl solution. Corrosion inhibition of stearamidine follows Langmuir-type adsorption isotherm, involving both physisorption and chemisorptions. It is observed that the maximum corrosion inhibition efficiency (96%) is achieved for 0.2 mM stearamidine in 3.0 wt.% NaCl solution at 300 K.

#### Data Availability

The data used to support the findings of this study are available from the corresponding author on request.

## Conflicts of Interest

The authors declare no conflicts of interest.

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