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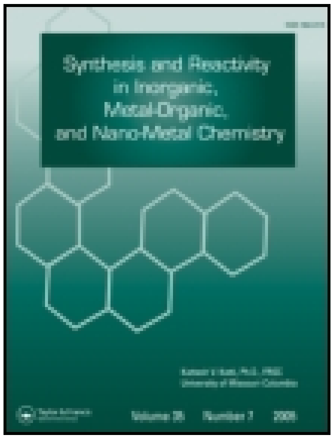

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Synthesis, Characterization, and Study the Inhibitory Effect of Thiazole and Thiadiazole Derivatives Toward the Corrosion of Copper in Acidic Media

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The authors describe the synthesis and characterization of two different five-member heterocycle derivatives (thiazole and thiadiazole) by several organic procedures. These compounds were characterized by FTIR, ^{1}H NMR, and elemental analyses. Also, the authors attempt to study the inhibition effects of these derivatives to the corrosion of copper in nitric acid media by weight loss technique. The temperature factor affecting the inhibition efficiency of these derivatives is discussed. Also, the inhibition efficiency depended on the changing substituents on the molecules. It was found from the results that the thiadiazole inhibitors have inhibition efficiency more than thiazole inhibitors.

Keywords: thiazole, thiadiazole, corrosion, Synthesis, inhibitory effect

Introduction

The use of heterocycles compounds to inhibit corrosion has assumed great significance due to their applications in preventing corrosion under various corrosion environments.[1,2] The interaction between these compounds and metallic surface is attributed by adsorption phenomenon. The structures of these compounds have a strong relationship in the inhibition process, and thus the effect of the functional group in adsorption processes varies because of the diversity of the substitutes on these compounds.[3]

Heteroatom derivatives with high electron density, which are considered as adsorption centers, are effective as corrosion inhibitors.[4–7] The compounds containing both nitrogen and sulfur atoms in their structures behaved as good inhibitors when we compared with those containing only one of them.[8]

In the literature, various thiazole and thiadiazole derivatives have been used as corrosion inhibitors for different metals and solutions and it has been found that these derivatives have good inhibition efficiency effects.[9–14] The inhibition properties of these heteroatoms compounds (thiazole and thiadiazole) are attributed to their molecular structures. The planarity and pairs of free electrons in heteroatoms are an important reason that makes the adsorption of these derivatives on the metal surface good, also these heterocyclic derivatives are considered as non-cytotoxic substances.[15] This environmentally friendly property makes them used as inhibitors to replacing some toxic organic inhibitors that are used in the same fields.[16]

This work was designed to study (a) corrosion inhibition of copper in nitric acid solution by some thiazole and thiadiazole derivatives using weight loss method, (b) the effect of the substituent groups in thiazole derivatives on the inhibition efficiency, (c) the difference in inhibition ability between thiazole and thiadiazole derivatives, and also (d) the effect of temperature on the corrosion rate.

Experimental

The inhibitors (**1–5**) were synthesized in the laboratory according to the following procedures. These compounds were characterized by FTIR, ^{1}H NMR, and elemental analysis. All chemicals and solvents were of reagent grade (Aldrich Chemicals Co.) and used without further purification. Infrared spectra were recorded with a Shimadzu 8000 FTIR spectrophotometer in the wave number range 4000–400 cm^{-1} with samples embedded in KBr disc. ^{1}H NMR spectra were obtained with Bruker spectrometer model Ultra Shield at 300 MHz. The compounds were dissolved in DMSO-d_6 solution with the TMS as internal standard. The CHNS data were recorded on EuRO EA model EA3000.

The aggressive solutions used were made of 69% HNO_3. Appropriate concentrations of acid were prepared using deionized water. The concentration range of inhibitor

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employed was 50–250 ppm in the 1M HNO_3 acid. The weight loss measurements were carried out in 30, 40, 50, and 60°C for 3 h using sheets of pure copper with dimensions (2×2.5 cm^2). Before the measurements, the copper specimens were polished successively with emery paper, degreased with acetone, washed with deionized water, and finally dried at room temperature. The coupons were weighted and immersed in 1M of HNO_3 solution with and without compounds **1–5**. At the end of the test, the coupons were taken out, washed with distilled water, degreased with ethanol, washed again with distilled water, dried, and then weighted again. The loss in weight, corrosion rate ($R_{corr.}$), the degree of the surface coverage (θ), and the percentage of inhibition efficiency (%IE) were calculated at different inhibitors concentrations according to the following equation:-

$$\Delta W = \frac{W_1 - W_2}{A} \tag{1}$$

$$R_{corr.} = \frac{\Delta W}{t} \tag{2}$$

$$\theta = \left[1 - \frac{W_{(W)}}{W_{(WO)}}\right] \tag{3}$$

$$\%IE = \left[1 - \frac{W_{(W)}}{W_{(WO)}}\right] \times 100 \tag{4}$$

Where, W_1 is the initial weight of the specimen (mg), W_2 is the specimen weight (mg) after the immersion period, A is the area of the specimen (cm^2), t is the immersion time (s), $R_{corr.}$ is the corrosion rate ($mg/cm^2.s$), and $W_{(w)}$ and $W_{(wo)}$ are weight loss of specimen with and without the inhibitors, respectively.

Preparation of Compounds 1–5

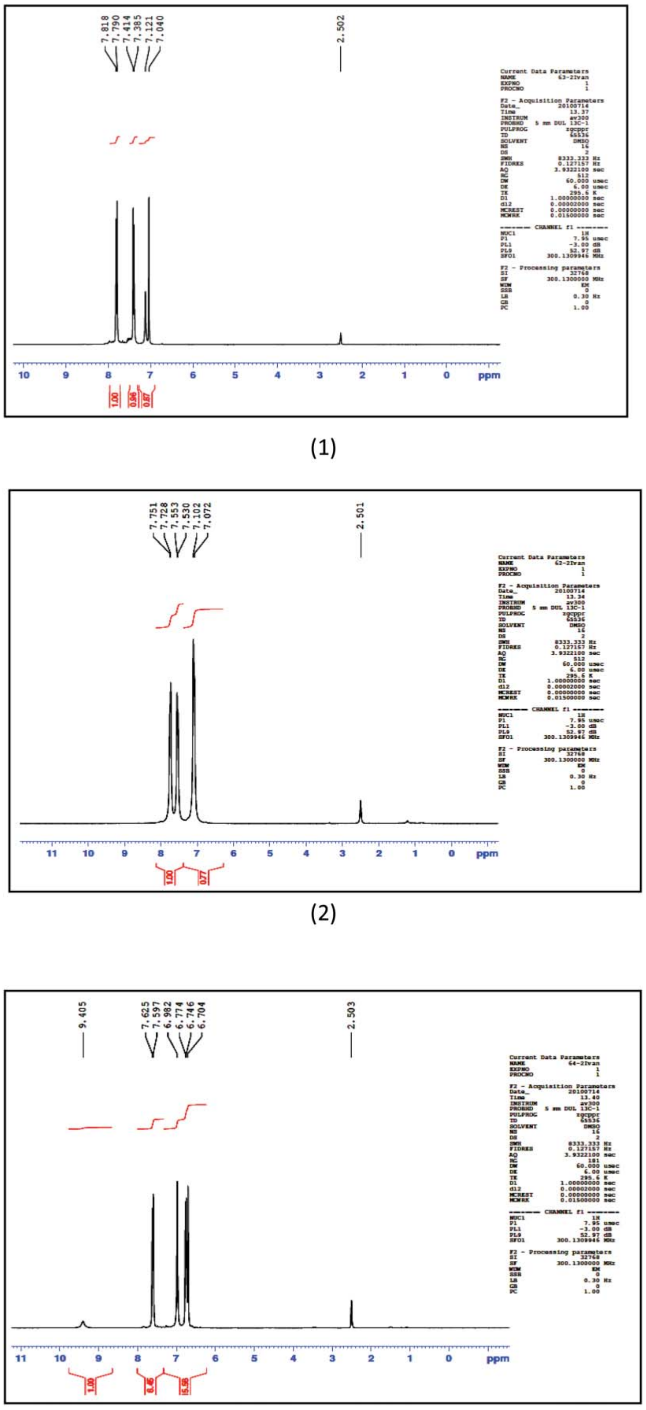
General procedure for preparation the compounds *1–3*

These compounds were prepared according to the procedure that was described in Siddiqui et al.[17] The title compounds were prepared by the addition of iodine (2.54 g, 0.01 mol) to the 4-substituted acetophenone (0.01 mol) and thiourea (1.52 g, 0.02 mol), followed by heating of the mixture overnight in an oil bath at 100°C. After cooling, the reaction mixture was washed with diethylether (100 mL) to remove any unreacted iodine and substituted acetophenone. The solid residue was put in (200 mL) of cold water and treated with 25% aqueous ammonium hydroxide (to pH 9–10). The precipitated thiazoles were collected and purified by crystallization from hot ethanol.

2-Amino-4-(4-chlorophenyl)-thiazole (1): Yield (81%); mp: 168–170°C; FTIR (KBr disk cm^{-1}): 3439, 3284 symm. and asymm. NH_2 group, 1633 (C = N); 1H NMR (DMSO-d_6, 300 MHz, δ): 7.81–7.79 (d, 2H, phenyl), 7.41–7.38 (d, 2H, phenyl), 7.12 (s, 2H, NH_2), 7.04 (s, 1H, thiazole ring); Anal. Calcd. for $C_9H_7ClN_2S$ (210.5 g/mol): C, 51.31; H, 3.33; N, 13.30; S, 15.20. Found: C, 51.55; H, 3.39; N, 13.50; S, 15.33.

2-Amino-4-(4-bromophenyl)-thiazole (2): Yield (84%); mp: 185–187°C; FTIR (KBr disk cm^{-1}): 3429, 3286 symm. and asymm. NH_2 group, 1627 (C = N); 1H NMR (DMSO-d_6, 300 MHz, δ): 7.75–7.72 (d, 2H, phenyl), 7.55–7.53 (d, 2H, phenyl), 7.10 (s, 2H, NH_2), 7.07 (s, 1H, thiazole ring); Anal. Calcd. for $C_9H_7BrN_2S$ (255 g/mol): C, 42.35; H, 2.75; N, 10.98; S, 12.55. Found: C, 42.61; H, 2.77; N, 11.05; S, 12.43.

4-(2-Aminothiazole-4-yl)-phenol (3): Yield (79%); mp: 208–210°C; FTIR (KBr disk cm^{-1}): 3447, 3304 symm. and asymm. NH_2 group, 3198 (O-H), 1616 (C = N); 1H NMR

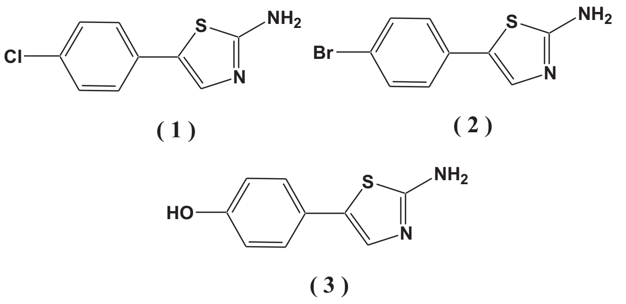


(1)

(2)

(3)

Fig. 1. ^{1}H NMR spectra of compounds **1**, **2**, and **3**.



Sch. 1. The chemical structures of compounds **1–3**.

(DMSO-d_6, 300 MHz, δ): 9.4 (br. 1H, phenolic OH), 7.62–7.59 (d, 2H, phenyl), 6.98 (s, 2H, NH_2), 6.77–6.74 (d, 2H, phenyl), 6.70 (s, 1H, thiazole ring); Anal. Calcd. for $C_9H_8N_2OS$ (192 g/mol): C, 56.25; H, 4.17; N, 14.58; S, 16.67. Found: C, 56.31; H, 3.99; N, 14.31; S, 16.42.

General procedure for preparation the compounds (4 and 5)

These compounds were prepared according to the modified procedure that was described in Tomi et al.[18] To a stirred solution of 2-amino-5-mercapto-1,3,4-thiadiazole (X) (1.00 g, 7.5 mmol) in (25 mL) of absolute ethanol, (0.42 g, 7.5 mmol) of potassium hydroxide that was dissolved in a minimum volume of water is added slowly. After heating the mixture for 15 minutes and cooling, (3.75 mmol) of 1,2-dibromoethane for compound **4** and 1,3-dibromopropane for compound **5** were added dropwise. The solution was refluxed for 5 h, and after cooling the mixture was poured into ice water. The precipitate was filtered and washed several times with water and ethanol then dried.

5,5′-(Ethane-1, 2-diyldisulfanediyl) bis (1,3,4-thiadiazole-2-amine) (4): Yield (73%); mp: 256–258°C; FTIR (KBr disk cm^{-1}): 3313, 3097 symm. and asymm. NH_2 group, 2943 (C-H) aliph.; 1631 (C = N); 1H NMR (DMSO-d_6, 300 MHz, δ): 7.32 (s, 4H, $2NH_2$), 3.49–3.52 (t, 4H, $2CH_2$); Anal. Calcd. For $C_6H_8N_6S_4$ (292 g/mol): C, 24.66; H, 2.74; N, 28.77; S, 43.84. Found: C, 24.52; H, 2.71; N, 28.33; S, 43.21.

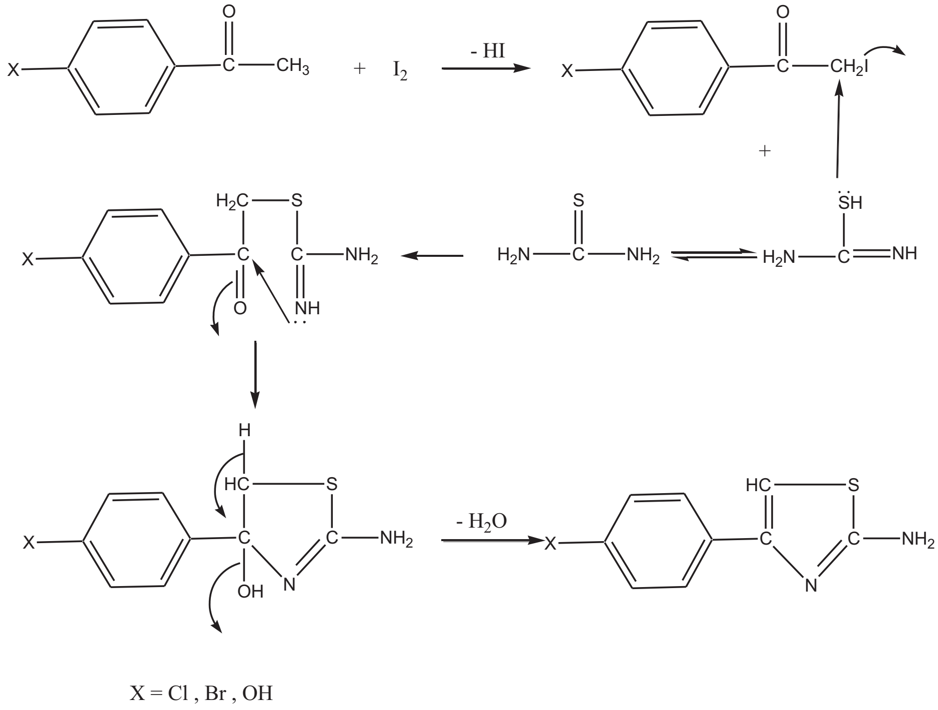
5,5′-(Propane-1,3-diyldisulfanediyl)bis(1,3,4-thiadiazole-2-amine) (5): Yield (77%); mp: 228–231°C; FTIR (KBr disk cm^{-1}): 3375, 3113 symm. and asymm. NH_2 group, 2933 (C-H) aliph.; 1602 (C = N); 1H NMR (DMSO-d_6, 300 MHz, δ): 7.30 (s, 4H, $2NH_2$), 3.14–3.16 (t, 4H, $2CH_2$ attached to thiadiazole ring), 1.99 (m, 2H, CH_2 center); Anal. Calcd. for $C_7H_{10}N_6S_4$ (306 g/mol): C, 27.45; H, 3.27; N, 27.45; S, 41.83. Found: C, 27.33; H, 3.17; N, 27.51; S, 41.92.

Results and Discussion

Synthesis

The aminothiazoles (**1–3**) (Scheme 1) were synthesized by the procedure that was described in Siddiqui et al.[17] The reaction between thiourea and 4-chloroacetophenone or 4-bromoacetophenone or 4-hydroxyacetophenone in the presence of iodine as oxidant agent yielded the aminothiazoles **1–3**. The suggested mechanism of this cyclization reaction may be outlined in the Scheme 2.

The structures of these compounds were confirmed by using elemental analysis and spectral (FTIR and ^{1}H NMR) data. These analyses are good evidence, which corresponds to the structures of the suggested compounds. The appearance of bands around 3430 and 3285 cm^{-1} in FTIR spectra



Sch. 2. The cyclization mechanism of compounds **1–3**.

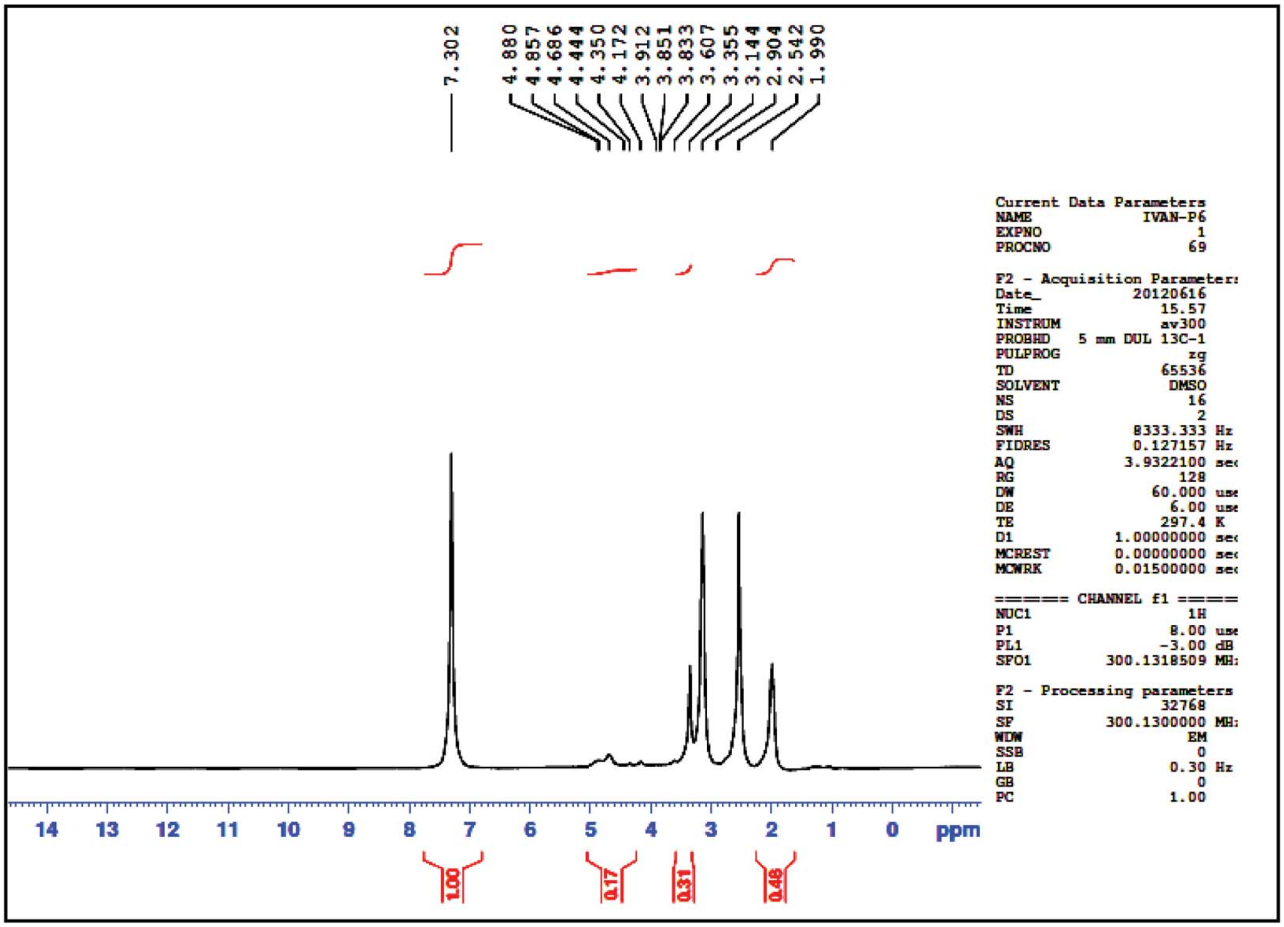
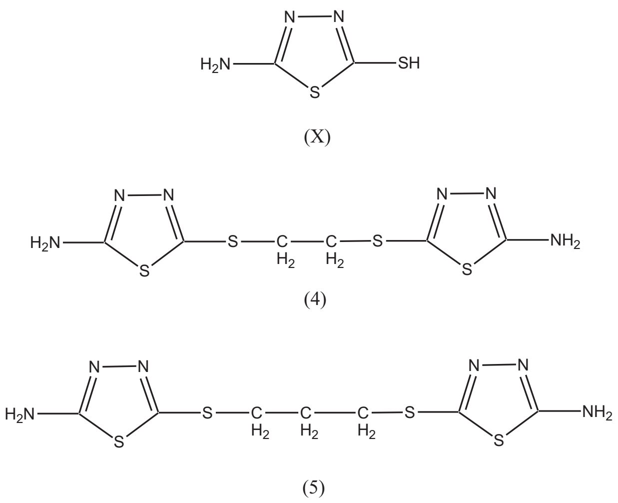


Fig. 2. ^{1}H NMR spectrum of compound **5**.

and peaks near 7.00 ppm in ^{1}H NMR spectra (Figure 1) that is assigned to the amino group on thiazole derivatives are good evidence for the structures given to the products. Also the band near 1620 cm^{-1} in FTIR spectra was utilized to confirm that cyclization reaction was occurring.



Sch. 3. The chemical structures of compounds **4** and **5**.

The aminothiadiazoles **4** and **5** (Scheme 3) were synthesized by thioetherification reaction between the compound (X) with 1,2-dibromo ethane and 1,3-dibromo propane in ethanolic KOH solution to product the compounds **4** and **5** respectively. The compound (X) was synthesized according to the procedure described in the reference.[19] Yield 88%, mp: 232°C; lit. mp: 232°C.[19]

The bands near 3300 and 3100 cm^{-1} in FTIR spectra and peaks near 7.30 ppm in ^{1}H NMR spectra (Figure 2) that are assigned to the NH_2 group are consistent with their proposed structures.

The microanalytical data of carbon, hydrogen, nitrogen and sulfur for compounds **1–5** were formed to be satisfactory and within the permissible limit error.

Weight Loss Measurement

Table 1 presents the values of percentage inhibition efficiency (%IE) and corrosion rate obtained from weight loss method at different concentrations of inhibitors at 30°C for 3 h. It has been found that all of these compounds inhibit the corrosion of copper in 1M HNO_3 solution, at all concentrations used in this study (i.e., 50–250 ppm), thus decreasing the weight loss of copper metal (Figure 3). The variation of

Table 1. Corrosion parameters for Cu in 1M HNO_3 solution in absence and presence of different concentrations of various inhibitors from weight loss measurements at 30°C for 3 h.

Inhibitor conc. (ppm)	Weight loss (mg)	%IE	$R_{corr.} \times 10^{-5}$ (mg / cm^2. s)
(1M) HNO_3	4.4821	—	4.15
1) $C_9H_7ClN_2S$			
50	1.9744	55.95	1.83
100	1.8861	57.92	1.75
150	1.7135	61.77	1.59
200	1.6118	64.04	1.49
250	1.5015	66.50	1.39
2) $C_9H_7BrN_2S$			
50	1.9515	56.46	1.81
100	1.8771	58.12	1.74
150	1.6659	62.83	1.54
200	1.5244	65.99	1.41
250	1.4204	68.31	1.32
3) $C_9H_8N_2OS$			
50	1.6919	62.25	1.57
100	1.4140	68.45	1.31
150	1.1958	73.32	1.11
200	0.9479	78.85	0.88
250	0.7947	82.27	0.74
4) $C_6H_8N_6S_4$			
50	0.8942	80.05	0.83
100	0.6929	84.54	0.64
150	0.5526	87.67	0.51
200	0.4294	90.42	0.40
250	0.2030	95.47	0.19
5) $C_7H_8N_6S_4$			
50	0.8552	80.92	0.79
100	0.6356	85.82	0.59
150	0.4912	89.04	0.45
200	0.3245	92.76	0.30
250	0.1135	97.46	0.11

inhibition efficiency to increase in inhibitor concentrations is shown in Figure 4. It has been indicated that the %IE for all of these compounds increases with the increase in concentration of inhibitor.

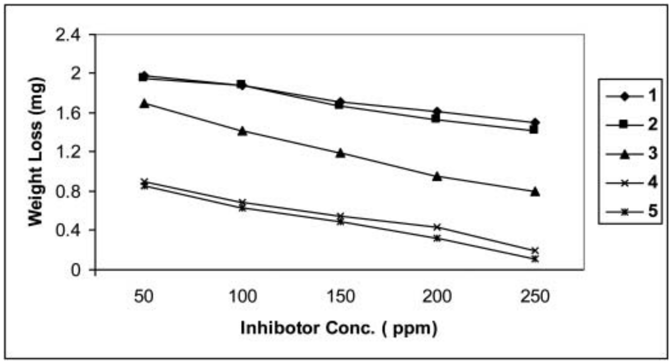


Fig. 3. Weight loss curves for copper in 1M HNO_3 in presence different concentrations of inhibitors **(1–5)** at 30°C for 3 h.

Adsorption Isotherm

In order to obtain a better understanding of corrosion inhibition of the metal surface, adsorption isotherms at 30°C were drawn. The degree of surface coverage (θ) for different inhibitor concentrations was evaluated from weight loss data. Data were tested graphically by fitting to various isotherms. A straight line was obtained by plotting log ($\theta/1-\theta$) versus log C (Figure 5) suggesting that the adsorption of the all organic compounds **1**–**5** from HNO_3 on copper surface follows Langmuir's adsorption isotherm (Eq. 5)[20,21]:

$$\log(\theta/1-\theta) = \log K + \log C \tag{5}$$

Where, K and C are the adsorptive equilibrium constant and the concentration of inhibitor, respectively.

Effect of the Temperature

The effect of rising temperature on the inhibition efficiency and the corrosion rate of Cu in 1M HNO_3 solution in the absence and presence of 250 ppm of the selected thiazole and thiadiazole compounds were studied of weight loss measurements over a temperature range from 30–60°C for 3 h. From Table 2 and Figure 6 it is clear that the corrosion inhibition decreases as the temperature as increases. This indicates that the corrosion inhibition takes place by adsorption of the inhibitors at the electrode solution interface.[22,23]

The activation energy (E_a) of the corrosion process was calculated using the Arrhenius equation[24]:

$$R_{corr.} = A\ e^{-Ea/RT} \tag{6}$$

And logarithmic form

$$\log R_{corr.} = \log A - Ea/2.303RT \tag{7}$$

Where A is Arrhenius constant, R is the general gas constant, and the T value is absolute temperature.

Figure 7 shows Arrhenius plots (log R_{corr} vs. 1/T) for uninhibited Cu in 1M HNO_3 and in the presence of the compounds **1**–**5** (Table 3). The values of E_a can be obtained from the slope of the straight lines. E_a found to be 98.82, 105.04, 114.56, 128.9, and 130.40 KJ/mol for compounds **1**–**5**, respectively, and for copper in 1M HNO_3 without the inhibitor is equal 76.18 KJ/mol, which is accordance with literature values.[22]

It is clear that the values of E_a increase in the presence of the inhibitors. This result is in accordance with the strength of adsorption on the copper surface[25] and indicates that the formation of the adsorption film occurs by a physical mechanism.[26] This was attributed to an appreciable decrease in the adsorption process of the inhibitor on the metal with increase of temperature and corresponding increase in the reaction

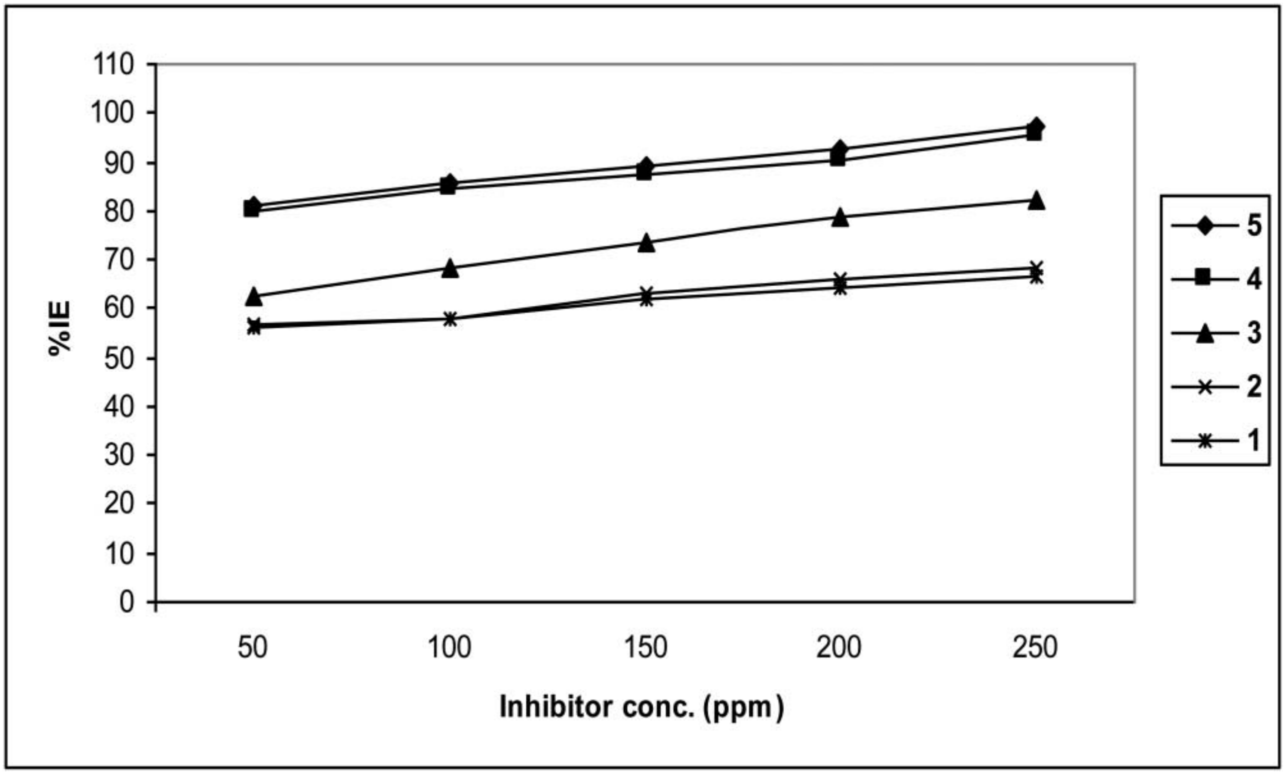


Fig. 4. Percentage inhibition efficiency (%IE) curves for copper in 1M HNO_3 in presence different concentrations of inhibitors **(1–5)** at 30°C for 3 h.

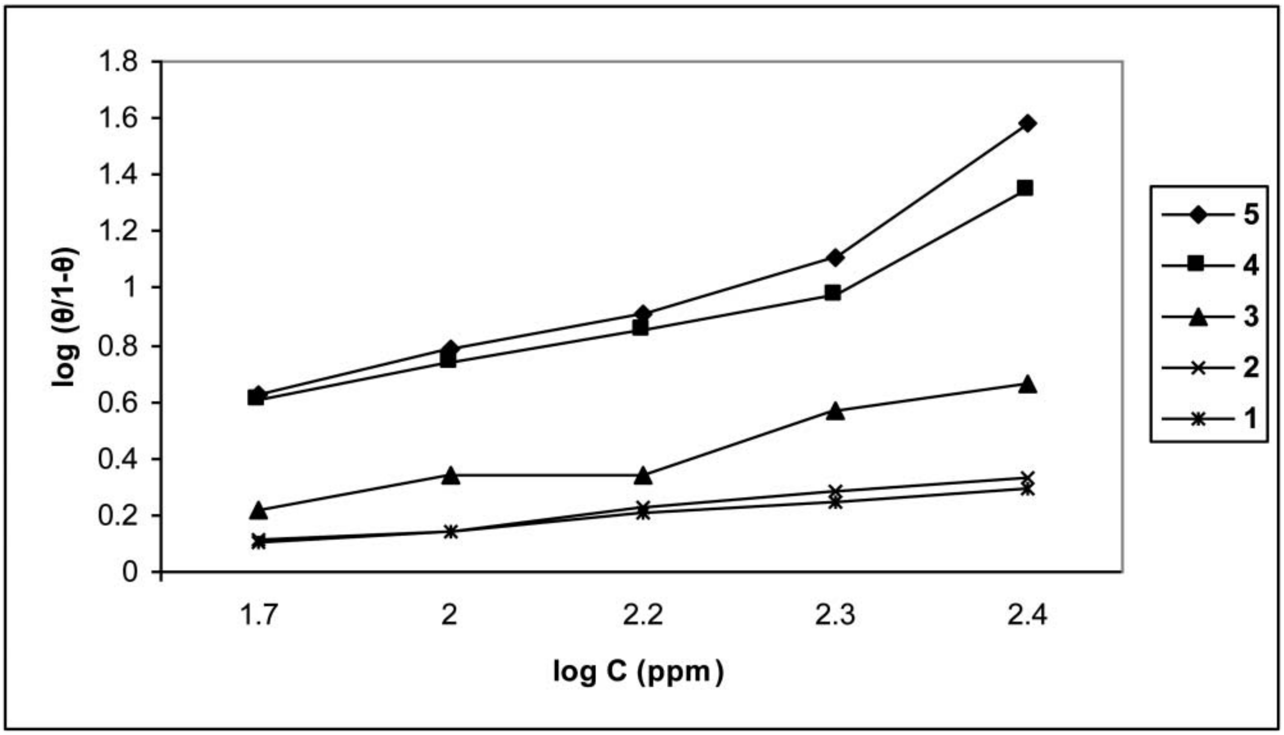


Fig. 5. Log($\theta/1-\theta$) vs. log C curves for copper in 1M HNO_3 in presence different concentrations of inhibitors **(1–5)** at 30°C for 3 h.

Table 2. Inhibition efficiency at 250 ppm of compounds (**1–5**) for the corrosion of Cu in 1M HNO_3 after 3h immersion at different temperature.

	Inhibition efficiency (IE)				
T°/C	1	2	3	4	5
30	66.50	68.31	82.27	95.47	97.46
40	45.01	51.03	55.94	57.54	58.89
50	18.72	20.57	35.43	39.18	41.19
60	5.69	2.28	14.18	18.72	19.8

Table 3. Variations of log rate without and with the presence of inhibitors verses reciprocal of temperature.

	Log $R_{coor.}$					
$1/T, 10^{-3}\,K^{-1}$	Copper	1	2	3	4	5
3.300	−4.38	−4.86	−4.88	−5.13	−5.74	−5.98
3.195	−3.92	−4.18	−4.20	−4.28	−4.30	−4.31
3.096	−3.52	−3.51	−3.62	−3.71	−3.74	−3.76
3.003	−3.15	−3.13	−3.18	−3.22	−3.24	−3.25

rate because of the greater area of the metal that is exposed to HNO_3 solution.[27]

Mechanism of Inhibition

The inhibition efficiency of thiazole and thiadiazole derivatives toward the corrosion of copper in 1M HNO_3 can be explained on the basis of the number of adsorption sites, their charge density, molecular size, mode of interaction with metal surface, and the ability to form metallic complexes.[27] It is apparent from the molecular structures that these compounds are able to get adsorbed on the metal surface through π-electrons of aromatic ring and lone pair of electrons on the N and S atoms, and as a protonated species such as amines.[20]

In this study, we compared the value of%IE at the limiting value and the corresponding inhibitor concentration. We concluded that the relative strength of these heterocycles compounds as inhibitors increases in the following order: **1**< **2** < **3**< **4**< **5**.

The derivatives **1–5** have either electron-donating groups such as OH group in the compound **3**, or electron withdrawing group such as Cl and Br in compounds **1** and **2**. The above order of %IE may be originated from the changing the substituents in the ring. It is clear from the above sequence that compounds containing electron donating groups are more efficient inhibitors than those containing electron withdrawing groups. While the compounds **4** and **5** give further improvement in the inhibition efficiency due to an increase in the number of active centers as well as the surface area. Both of inhibitors have four S and six N atoms as active centers.

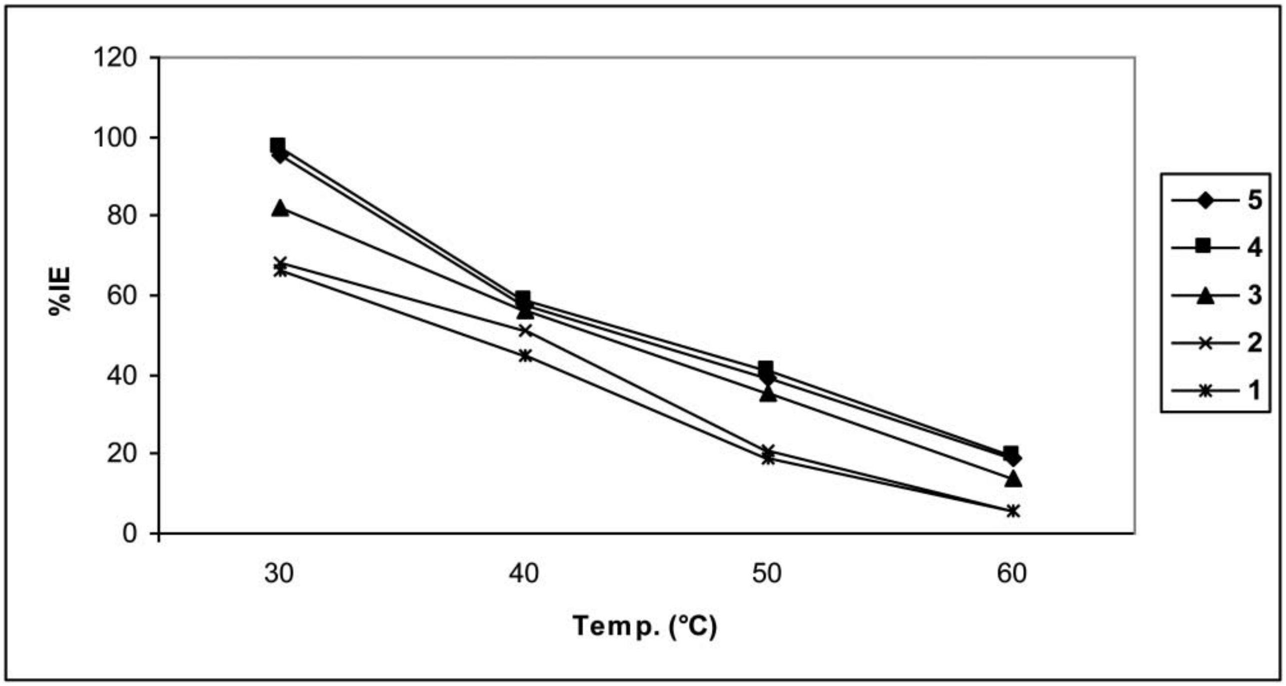


Fig. 6. Percentage inhibition efficiency (%IE) curves for copper in 1M HNO_3 in presence different concentrations of inhibitors **(1–5)** at varying temperatures for 3 h.

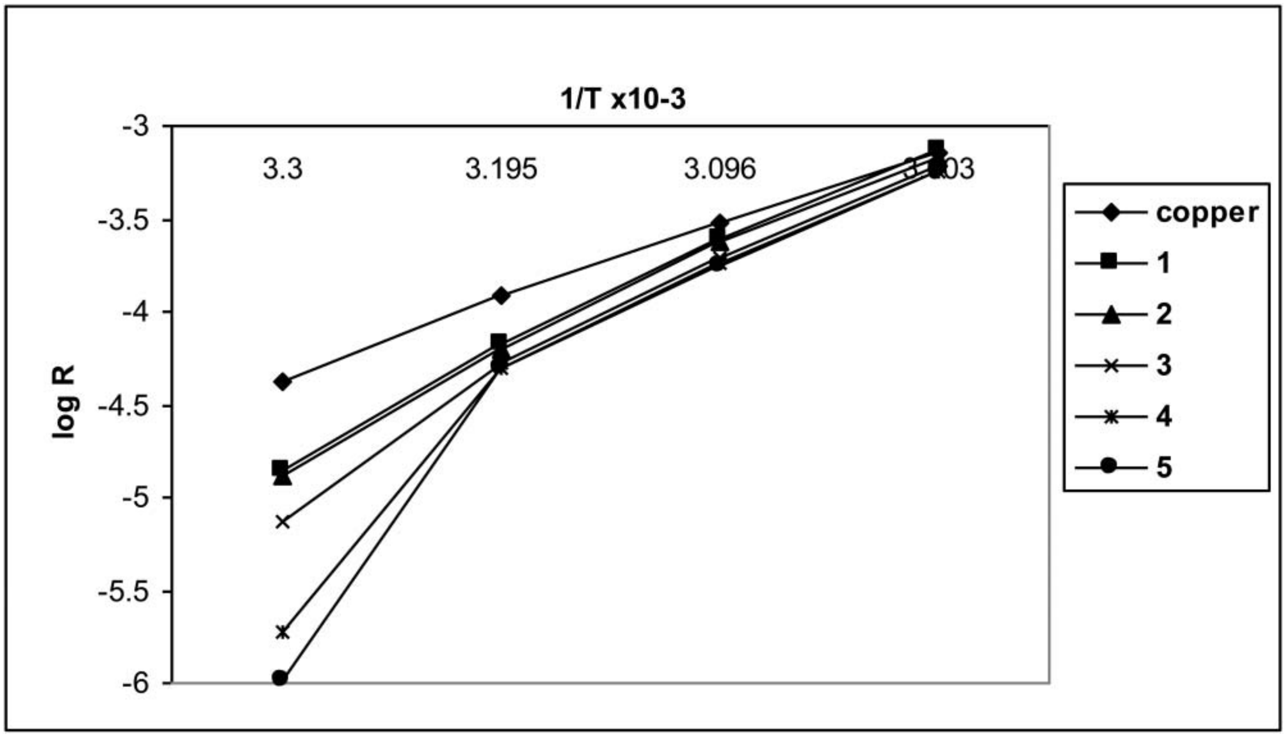


Fig. 7. Arrhenius plots of copper in 1M HNO_3 with and without different inhibitors **(1–5)**.

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