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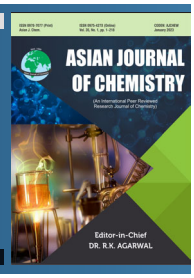

Synthesis, Antimicrobial and Corrosion Inhibition Studies of 1,3-Benzothiazole Derivatives

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With an emphasis on potential medicinal and corrosion inhibition applications, four *N*-(4-(substituted benzylidene)benzo[*d*]thiazol-2-amine were synthesized by the condensation of 2,3-dihydro-1,3-benzothiazol-2-amine with 4-substituted benzaldehydes. Nuclear magnetic resonance (NMR), infrared (IR) and mass spectroscopy were used to characterize the synthesized compounds. To assess the antibacterial activity of synthetic drugs, the conventional serial dilution approach has been used. The synthesized compounds showed excellent bioactivities comparable to those of a typical medication. The corrosion inhibition effect of benzothiazole derivatives has been investigated against mild steel in 0.1 N HCl solution using potentiodynamic polarization and electrochemical impedance spectroscopy methods at room temperature. The results revealed that 4-{[(1,3-benzothiazol-2-yl)imino]methyl}-*N*,*N*-dimethylaniline inhibitor has high inhibition efficiency (56.39%) at room temperature for a 50 mg/L concentration when compared to other compounds.

Keywords: Benzothiazole, Corrosion inhibitor, Mild steel, Potentiodynamic polarization, Antimicrobial activity.

INTRODUCTION

In benzothiazole, a benzene ring and a thiazole ring are fused together. The compound benzothiazole has a significant impact on medicinal chemistry and a wide spectrum of biological activities containing anticancer [1,2], antibacterial [3,4], antituberculosis [5,6], antidiabetic [7], anthelmintic [8], antitumor [9-11], antiviral [12,13], antioxidant [14], antiglutamate and antiparkinsonism [15], anti-inflammatory [16,17], anticonvulsant [18], muscle relaxant activities [19], neuroprotective [20], inhibitors of several enzymes and so on [21].

Mild steel is the most prevalent type of steel, owing to its low cost and the fact that it has vital and beneficial qualities in petroleum, industrial and medical uses. In addition, the low corrosion resistance of mild steel, particularly in acidic and basic conditions, is a challenge for corrosion scientists and researchers [22]. Even though many of them are expensive and dangerous, synthetic organic inhibitors are commonly used. The researchers made the decision to concentrate their efforts on developing novel ecologically benign (or non-toxic) degradation inhibitors since they are the most practical substitute for toxic and inorganic inhibitors and are also practical from a financial and environmental standpoint. It has been demanded that some synthetic organic compounds can serve as effective corrosion inhibitors and are safe for the environment [23-27]. In the process of acid pickling and industrial acid cleaning, these chemicals will be significantly dissolved. There will be material losses as well as electrochemical and chemical etching. Since, it is less expensive than other mineral acids and presents no issues in industrial settings, hydrochloric acid is commonly used [28]. According to the literature, organic compounds containing unsaturated bonds, aromatic rings and electronegative oxygen, sulphur, nitrogen and phosphorous atoms make up the majority of corrosion inhibitors [29,30].

As a result of their important biological activities and corrosion inhibitor capabilities, the synthesis of benzothiazoles is of great interest. In this study, the reaction between amino benzothiazoles with substituted aldehydes were conducted to evaluate their antimicrobial activities as well as their corrosion inhibition. The corrosion inhibition of the synthesized compounds (**3a-d**) on mild steel in 0.1 M HCl was studied by Tafel polarization and electrochemical impedence spectroscopy methods.

EXPERIMENTAL

Without further purification, chemicals procured from Merck, USA were used. Thin-layer chromatography was used

to keep track of every response. The melting points were calculated using an uncorrected Bio-Techniques India (BTI-39) instrument. The IR spectra were captured in KBr using Bruker Alpha FT-IR spectrophotometer. On an Agilent NMR (400 MHz) spectrometer, ^{1}H & ^{13}C NMR spectra were captured using a solvent DMSO and internal standard is TMS. A Waters Synapt G2 mass spectrometer was used to record the mass spectra.

Synthesis of *N*-(4-(substituted benzylidene)benzo[*d*]-thiazol-2-amine (3a-d): 4-Substituted benzaldehyde (**1**) (0.01 mol) dissolved in ethanol was added to benzo[*d*]thiazol-2-amine (**2**) (0.01 mol) followed by the addition of 2 drops of conc. H_2SO_4 and then allowed to reflux for 5 h. After cooling, the solid product underwent filtering and purification *via* recrystallization from ethanol (**Scheme-I**).

***N*-Benzylidenebenzo[*d*]thiazol-2-amine (3a):** White, yield: 64%; m.p.: 199-201 ºC. FT-IR (KBr, ν_{max}, cm^{-1}): 1645.13 (CH=N *str.*), 3075 (Ar-CH *str.*). ^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 9.26 (s, 1H, CH=N proton), 7.53-8.23 (m, 6H, phenyl H-2, H-6 and benzothiazole protons), 6.96-7.48 (m, 3H, phenyl H-3, H-4 and H-5 protons). ^{13}C NMR ($CDCl_3$, 400 MHz, δ ppm): 172.4 (benzothiazole C-2), 147.6 (benzothiazole C-4), 121.6 (benzothiazole C-5), 124.3 (benzothiazole C-6), 123.5 (benzothiazole C-7), 120.7 (benzothiazole C-8), 123.8 (benzothiazole C-9), 159.0 (CH=N), 135.2 (phenyl C-1), 128.3 (phenyl C-2 and C-6), 128.7 (phenyl C-3 and C-5) and 131.5 (phenyl C-4). Mass spectrum (m.f.: $C_{14}H_{10}N_2S$, *m.w.*: 238): molecular ion peak, *m/z* 239 (M^++ 1) .

4-((Benzo[*d*]thiazol-2-ylimino)methyl)phenol (3b): Yellow, yield: 70%; m.p.: 192-194 ºC. FT-IR (KBr, ν_{max}, cm^{-1}): 1638.11 (CH=N *str.*), 3376 (O-H *str.*). ^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 9.26(s, 1H, CH=N proton), 7.15-7.80 (m, 6H, phenyl H-2, H-6 and benzothiazole protons), 6.93-6.95 (d, 2H, phenyl H-3 and H-5 protons), 5.65 (s, 1H, OH proton). ^{13}C NMR ($CDCl_3$, 400 MHz, δ ppm): 172.4 (benzothiazole C-2), 147.6 (benzothiazole C-4), 121.6 (benzothiazole C-5), 124.3 (benzothiazole C-6), 123.5 (benzothiazole C-7), 120.7 (benzothiazole C-8), 123.8 (benzothiazole C-9), 159.0 (CH=N), 128.2 (phenyl C-1), 129.7 (phenyl C-2 and C-6), 115.4 (phenyl C-3 and C-5) and 158.8 (phenyl C-4). Mass spectrum (m.f.: $C_{14}H_{10}N_2OS$, *m.w.*: 254): molecular ion peak, *m/z* 255 (M^++1).

***N*-(4-bromobenzylidene)benzo[*d*]thiazol-2-amine (3c):** White, yield: 68%; m.p.: 202-204 ºC. FT-IR (KBr, ν_{max}, cm^{-1}): 1635 (CH=N *str.*), 714 (C-Br *str.*). ^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 9.26 (s, 1H, CH=N proton), 7.53-8.28(m, 6H, phenyl H-2, H-6 and benzothiazole protons), 7.13-7.35 (d, 2H, phenyl H-3 and H-5 protons). ^{13}C NMR ($CDCl_3$, 400 MHz, δ ppm): 172.4 (benzothiazole C-2), 147.6 (benzothiazole C-4), 121.6 (benzothiazole C-5), 124.3 (benzothiazole C-6), 123.5 (benzothiazole C-7), 120.7 (benzothiazole C-8), 123.8 (benzothiazole C-9), 159.0 (CH=N), 134.6 (phenyl C-1), 128.4 (phenyl C-2 and C-6), 131.6 (phenyl C-3 and C-5) and 125.5 (phenyl C-4). Mass spectrum (m.f.: $C_{14}H_9N_2SBr$, *m.w.*: 317): molecular ion peak, *m/z* 319 (M^++ 2)

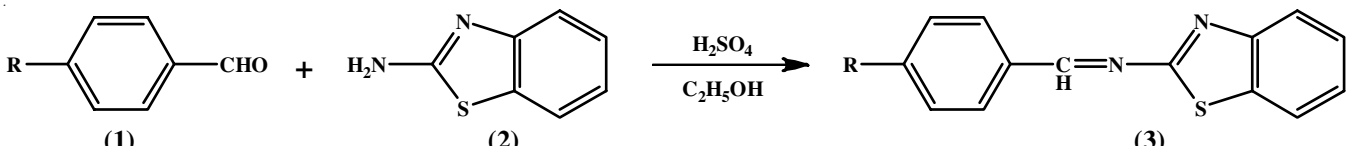
***N*-(4-(Dimethylamino)benzylidene)benzo[*d*]thiazol-2-amine (3d):** White, yield: 67%; m.p.: >300 ºC. FT-IR (KBr, ν_{max}, cm^{-1}): 1633 (CH=N *str.*), 1573 (*N*,*N*-dimethyl *str.*). ^{1}H NMR ($CDCl_3$, 400 MHz, δ ppm): 9.26 (s, 1H, CH=N proton), 3.14 (s, 6H, -N(CH_3)$_2$, 7.15-8.30 (m, 6H, phenyl H-2, H-6 and benzothiazole protons), 6.93-6.95 (d, 2H, phenyl H-3 and H-5 protons). ^{13}C NMR ($CDCl_3$, 400 MHz, δ ppm): 172.4 (benzothiazole C-2), 147.6 (benzothiazole C-4), 121.6 (benzothiazole C-5), 124.3 (benzothiazole C-6), 123.5 (benzothiazole C-7), 120.7 (benzothiazole C-8), 123.8 (benzothiazole C-9), 159.0 (CH=N), 125.2 (phenyl C-1), 128.1 (phenyl C-2 and C-6), 110.8 (phenyl C-3 and C-5), 154.1 (phenyl C-4) and 40.8 (N(CH_3)$_2$). Mass spectrum (m.f.: $C_{16}H_{15}N_3S$, *m.w.*: 281): Molecular ion peak, *m/z* 282 (M^++1).

Antimicrobial activity: The antibacterial activity of the synthesized N-(4-(substituted benzylidene)benzo[*d*]thiazol-2-amine was evaluated using the serial dilution method to determine the least inhibitory concentration (MIC) [22].

Corrosion inhibition studies

Preparation of specimen and electrolyte preparation: As working electrode, a mild steel specimen with an actual size of 1 cm × 1 cm was used. The samples were removed from the work area after being covered in epoxy resin. Acetone was used to clean the specimens' surfaces, which were then dried and used for examination after being cleansed with distilled water and acetone. Different grades of emery paper (100, 400, 800 and 1200) were employed for the surface preparation. By dilution of 37% HCl (Merck), which is utilized as corrosive medium, with distilled water, electrolyte of 0.1 M HCl solution was prepared. To prepare the inhibitor solutions, 50 mg/L of inhibitor (**3a-d**) was dissolved in 0.1 M HCl acid solution.

Corrosion analysis: Electrochemical investigations with and without inhibitors were conducted using a potentiostat (CH-instrument beta software). The three-electrode glass cell was used for both potentiodynamic polarization and electrochemical impedance spectroscopy, with mild steel specimen, platinum and calomel acting as working, counter and reference electrodes, respectively. The polished mild steel specimen was subjected to a corrosive media (1 cm^2) of 0.1M HCl at room temperature (30 ºC), allowing the development of a steady state open circuit potential both in the presence and absence of inhibitors (OCP). To record the Tafel (I-E) plots, potentiodynamic polarization experiments were performed from OCP at a scan rate of 1 mV/s^{-1} in a potential range of ± 200 mV. The inhibition efficiency (% IE) was computed from the corrosion potential (E_{corr}), corrosion current density (i_{corr}) and corrosion



Scheme-I

rates were measured. In order to conduct the EIS investigations, an OCP was impressed with an AC signal of 10 mV amplitude and a frequency range of 100 kHz to 10 mHz.

RESULTS AND DISCUSSION

Antibacterial activity: The newly synthesized compounds **3a-d** were tested for their antibacterial efficacy against four distinct pathogens, including *Escherichia coli*, *Staphylococcus aureus*, *Pseudomonas aeruginosa* and *Klebsiella pneumonia*. The standard drug was ampicillin. Due to the inclusion of -Br and -OH substituents, compounds **3a** and **3b** demonstrated good antibacterial activity comparable to that of the standard. Table-1 contains the minimal inhibitory concentrations (MIC) for the synthesized compounds.

Antifungal activity: Using the serial dilution approach, the antifungal activity of each produced series was evaluated against *Penicillium maneffei*, *Trichophyton mentagrophytes*, *Aspergillus flavus* and *Aspergillus fumigates.* The standard drug used was itraconozole. Among the examined compounds, compound **3c** containing -Br demonstrated strong antifungal activity, whereas the other compounds displayed antifungal activity that was moderately equal to the standard. For each of the produced series, the minimum inhibitory concentration (MIC) was provided in Table-1.

Potentiodynamic polarization study: The potentiodynamic polarization results showed that the synthesized compounds **3a-d** provided the protection on the mild steel 0.1 N HCl solution. The best performance is observed in the potentiodynamic polarization plots (Fig. 1) at the ambient conditions. The corrosion rate (CR) of mild steel was more when compared with the inhibitors [31] (Table-2). Compound **3d** shows the highest inhibition effectiveness (IE 56.39 %) when compared to different inhibitors and hence higher corrosion resistance. The corrosion behaviour is caused by the inhibitor adsorption on the compounds at the mild steel/solution interface and thus, compound **3d** shows the highest efficiency of the inhibitor at room temperature. Mild steel showed the cathodic corrosion potential when compared to a blank. Both the cathodic and anodic processes are suppressed, according to the potentiodynamic polarization curves (Fig. 1). Because of this, the inhibitor has a mixed type of action [32,33].

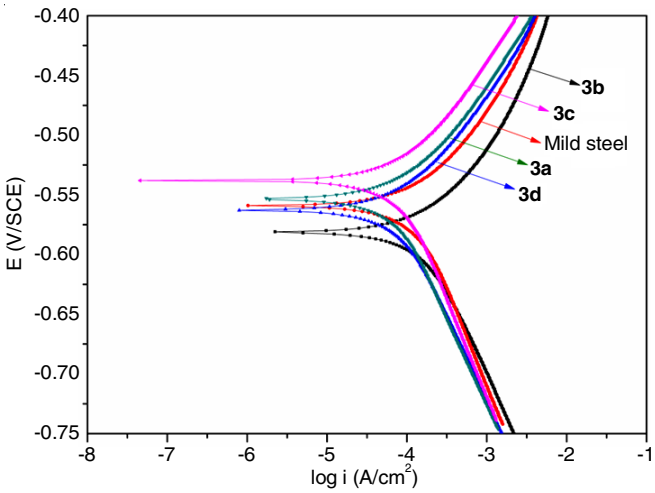


Fig. 1. Potentiodynamic polarization plots for MS in 0.1 M HCl with inhibitors (**3a-d**) and without inhibitors

Electrochemical impedance analysis: The divergence from perfect semicircular Nyquist plots indicates a rough and non-homogeneous surface, inhibitor adsorption or the creation of a porous layer. The charge transfer mechanism is supported by the form of impedance spectrum. Capacitive and inductive loops should have larger magnitudes for effective inhibition, as shown in the current experiment [34,35]. The Nyquist curves of mild steel in 0.1 M HCl in the presence and absence of inhibitors are shown in Fig. 2 at 30 ºC. Without inhibitor the mild steel shows smaller impedance module and shows larger corrosion rate (Table-2). But with the synthesized inhibitors (**3a-d**), radius of impedance module increased as change of inhibitors and shows better corrosion inhibition efficiency (%IE) (Table-2). The depression in Fig. 2 represents the metal

TABLE-1
ANTIMICROBIAL ACTIVITY OF N-(4-(SUBSTITUTED BENZYLIDENE)BENZO[*d*]THIAZOL-2-AMINES (**3a-d**)

Compound	MIC (µg/mL)							
	Antibacterial activity				Antifungal activity			
	E. coli	*S. aureus*	*P. aeroginosa*	*K. pneumonia*	*P. marneffei*	*T. mentagrophytes*	*A. flavus*	*A. fumigatus*
3a	6.25	12.5	12.5	12.500	6.25	12.5	12.5	12.500
3b	**3.125**	6.25	6.25	**3.125**	6.25	6.25	6.25	**3.125**
3c	**3.125**	6.25	**3.125**	**3.125**	**3.125**	**3.125**	**3.125**	**3.125**
3d	6.25	6.25	12.5	6.250	6.25	12.5	12.5	6.250
Ampicillin	6.25	6.25	6.25	6.250	–	–	–	–
Itraconozole	–	–	–	–	6.25	6.25	6.25	6.250

TABLE-2
POTENTIODYNAMIC POLARIZATION STUDIES ON MILD STEEL IN 0.1 M HCl SOLUTION WITH AND WITHOUT OF INHIBITORS

Compound	i_{corr} (µA/dm^2)	E_{corr} (V)	βc (mV/dec)	βa (mV/dec)	CR (mm/y)	IE (%)
Mild steel	207.00	-0.5544	7.260	8.602	2.519	–
3a	99.28	-0.5379	6.076	10.369	0.583	52.03
3b	98.30	-0.5603	6.161	9.444	0.983	52.51
3c	95.15	-0.5802	6.753	10.398	0.559	54.03
3d	90.29	-0.5631	7.204	10.684	0.508	56.39

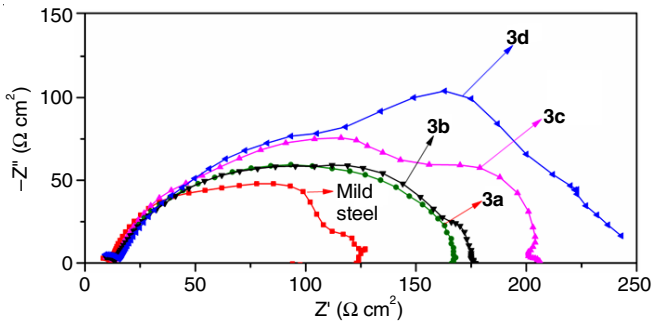


Fig. 2. Electrochemical impedance plot of MS in 0.1 M HCl with (**3a-d**) and without inhibitor

surface homogeneities that occured during the corrosion. The diameter of the capacitive loop shows the corrosion resistance and it can be seen that the resistance decreases a lot as the diameter of the capacitive loop declines.

Conclusion

A series of *N*-(4-(substituted benzylidene)benzo[*d*]thiazol-2-amine were synthesized by the condensation of 2,3-dihydro-1,3-benzothiazol-2-amine with 4-substituted benzaldehyde and characterized using the mass, NMR and IR spectroscopic techniques. These compounds exhibited potent antimicrobial activity comparable with the standard drugs. Moreover, 1,3-thiazole derivatives were found to be effective as mild steel corrosion inhibitors in 0.1 M HCl solution. According to the polarization curves, these compounds functioned as mixed-type inhibitors.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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