Research Article

Synthesis and Antimicrobial Activity of Some New Thiazolidin-4-one Derivatives of 4-(6-methylbenzo[d]thiazol-2-yl)benzamine

Awaz Jamil Hussein and Hashim Jalal Azeez

Department of Chemistry, College of Education-Scientific Department, Salahaddin University, Hawler, Kurdistan, Arbil, Iraq

Correspondence should be addressed to Awaz Jamil Hussein; awazjhusen@yahoo.com

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A number of derivatives of 2-(substituted phenyl)-3-(4-(6-methylbenzo[d]thiazol-2-yl)phenyl) thiazolidin-4-one (3a-j) have been synthesized from the reaction of 4-(6-methylbenzo[d]thiazol-2-yl)benzenamine(1), with different substituted benzaldehydes (2a-j), followed by cyclocondensation reaction of the prepared imines with 2-mercaptoacetic acid in high yields. Furthermore, the structures of the newly synthesized compounds were confirmed by FT-IR, ^{13}C -NMR, ^{13}C -DEPT, and ^1H -NMR spectral data. The imines and thiazolidin-4-one derivatives were evaluated for their antibacterial activity against *Escherichia coli* as gram negative and *Staphylococcus aureus* as gram positive, the results have shown significant activity against both types of bacteria.

1. Introduction

Imines can easily be prepared from amines and benzaldehydes [1, 2] and are useful precursor for the synthesis of different heterocyclic compounds like azetidinones [3, 4] and thiazolidin-4-ones [5–7]. Thiazolidin-4-ones are one of the most intensively investigated classes of aromatic heterocycles, containing sulfur and nitrogen in five-membered rings, these compounds can be prepared from imines and 2-mercaptoacetic acid [8]. Both imines and thiazolidin-4-one are known to possess impressive biological activities such as antibacterial [9, 10], antihyperglycemia [11], antifungal [12, 13], antagonist [14], insecticidal [15], anticonvulsant [16], and antitubercular agents [17]. Therefore it was envisaged that compounds containing both thiazole and thiazolidin-4-one moieties would have interesting biological activities [18]. In the present work, we have described the synthesis of some new thiazolidin-4-one compounds derived from 4-(6-methylbenzo[d]thiazol-2-yl)benzenamine, evaluating the effect of electron donating and electron withdrawing substituents, the biological activity and their spectroscopic studies were also investigated.

2. Experimental

Melting points were determined using an electrothermal melting point apparatus. IR spectra were recorded on a Bio-Rad Merlin FT-IR spectroscopy Mod FTS 3000, using KBr disc. ^1H -NMR and ^{13}C -NMR and ^{13}C -DEPT-135 spectra were recorded on a Bruker (300 MHz) with TMS as internal reference.

2.1. Synthesis of Imines: (Substituted benzylidene)-4-(6-methylbenzo[d]thiazol-2-yl)benzenamine (2a-j) [19]. According to the modified procedure, imines (2a-j) were synthesized by dissolving (2.4 g, 0.01 mol) of 4-(6-methylbenzo[d]thiazol-2-yl)benzenamine (1) in 96% ethanol (20 mL), and mixed with the solution of an appropriate substituted benzaldehyde (0.01 mol) in 96% ethanol (10 mL) with a few drops of acetic acid. The mixture was refluxed for (1–3 hrs) until the formation of imines which was monitored by TLC and formation of solid products. The cooled mixture was filtered, dried, and purified by hot ethanol to give pure crystals of compounds (2a-j).

FIGURE 1: ^1H -NMR spectrum of compound (2g). FIGURE 2: ^{13}C -NMR spectrum of compound (2g).

- 2a: R: H, $\text{C}_{21}\text{H}_{16}\text{N}_2\text{S}$, mp. 194–196°C, yield: 80%. FT-IR (KBr cm^{-1}): 3016, 2912, 2885, 1624, 1565, 1500, 1481.
- 2b: R: 4-Cl, $\text{C}_{21}\text{H}_{15}\text{ClN}_2\text{S}$, mp. 226–228°C, yield: 84%. FT-IR (KBr cm^{-1}): 3020, 2913, 2863, 1624, 1580, 1562, 1483.
- 2c: R: 3-Cl, $\text{C}_{21}\text{H}_{15}\text{ClN}_2\text{S}$, mp. 206–208°C, yield: 85%. FT-IR (KBr cm^{-1}): 3017, 2910, 2880, 1628, 1605, 1595.
- 2d: R: 2- NO_2 , $\text{C}_{21}\text{H}_{15}\text{N}_3\text{O}_2\text{S}$, mp. 208–210°C, yield: 80%. FT-IR (KBr cm^{-1}): 3022, 2913, 2881, 1615, 1607, 1580, 1512, 1377.

FIGURE 3: DEPT-135 spectrum of compound (2g).

- 2e: R: 2-F, $\text{C}_{21}\text{H}_{15}\text{FN}_2\text{S}$, mp. 139–141°C, yield: 83%. FT-IR (KBr cm^{-1}): 3023, 2924, 2854, 1627, 1603, 1590.
- 2f: R: 4- NO_2 , $\text{C}_{21}\text{H}_{15}\text{N}_3\text{O}_2\text{S}$, mp. 223–224°C, yield: 88%. FT-IR (KBr cm^{-1}): 3019, 2916, 2865, 1629, 1599, 1510, 1512, 1340.
- 2g: R: 4- OCH_3 , $\text{C}_{22}\text{H}_{18}\text{N}_2\text{OS}$, mp. 188–190°C, yield: 75%. FT-IR (KBr cm^{-1}): 3016, 2910, 1609, 1570, 1251, 1028; ^1H -NMR 2.51 (s, 3H, CH_3), 3.9 (s, 3H, $-\text{OCH}_3$), 7.02–8.1 (m, 11H, for aromatic ring, 8.44 (s, 1H, imine proton $-\text{CH}=\text{N}-$); ^{13}C -NMR: 21.55: (CH_3); 55.46: ($\text{O}-\text{CH}_3$); 114.2: $\text{C}_{2',2}$; 121: C_{12} ; 121.5: C_{15} ; 122.5: $\text{C}_{7,7'}$; 127.8: C_{14} ; 128.41: C_4 ; 129.00: $\text{C}_{8,8'}$; 130.79: $\text{C}_{3,3'}$; 135.2: C_9 ; 138: C_{13} ; 139: C_{11} ; 152.37: C_{16} ; 154.54: C_6 ; 160.28: C_5 ; 162.6: C_1 ; 165.5: C_{10} ; DEPT-135: 21.55: CH_3 ; 55.46: $-\text{OCH}_3$; 114.2: $\text{C}_{2',2}$; 121.3: C_{12} ; 121.5: C_{15} ; 122.5: $\text{C}_{7,7'}$; 127.8: C_{14} ; 129.00: $\text{C}_{8,8'}$; 160.28: C_5 .
- 2h: R: 4- CH_3 , $\text{C}_{22}\text{H}_{18}\text{N}_2\text{S}$, mp. 193–195°C, yield: 74%. FT-IR (KBr cm^{-1}): 3026, 2914, 2856, 1629, 1593, 1568, 1477.
- 2i: R: $-\text{CHO}$, $\text{C}_{22}\text{H}_{16}\text{N}_2\text{OS}$, mp. 225–227°C, yield: 80%. FT-IR (KBr): 3024, 2916, 2852, 2738, 1697, 1605, 1562, 1479; ^1H -NMR; 2.52 (s, 3H, CH_3), 10.1 (s, 1H, $-\text{CHO}$), 7.2–8.1 (m, 11H, for aromatic rings), 8.6 (s, 1H, imine proton $-\text{CH}=\text{N}-$); ^{13}C -NMR: 21.56 (CH_3); 121.36: C_{12} ; 121.58: C_{15} ; 122.68: $\text{C}_{7',7}$; 128.05: C_{14} ; 128.5: $\text{C}_{8,8'}$; 129.45: $\text{C}_{3',3}$; 130.07: $\text{C}_{2,2}$; 132.14: C_9 ; 135.45: C_{13} ; 138.23: C_{11} ; 140.9: $\text{C}_{1,4}$; 153.3: C_{16} ; 159.4: C_5 ; 164.0: C_{10} ; 191.70: CHO ; DEPT-135: 21.56 (CH_3); 121.36: C_{12} ; 121.58: C_{15} ; 122.68: $\text{C}_{7',7}$; 128.05: C_{14} ; 128.5: $\text{C}_{8,8'}$; 129.45: $\text{C}_{3',3}$; 130.07: $\text{C}_{2',2}$; 159.4: C_5 ; 191.7: CHO .

R: H, 4-Cl, 3-Cl, 2-NO₂, 3-NO₂, 4-NO₂, 2-F, 4-CH₃, 4-OCH₃, 4-CHO

SCHEME 1

2j: R: 3-NO₂, C₂₁H₁₅N₃O₂S, mp. 201–203°C, yield: 81%.
FT-IR (KBr cm⁻¹): 3019, 2920, 2887, 1625, 1597, 1566, 1519, 1342.

2.2. Synthesis of Thiazolidin-4-ones: 3-(4-(6-Methylbenzo[d]thiazol-2-yl)phenyl)-2-(4-substitutedphenyl) Thiazolidin-4-ones (3a-j) [20]. According to the modified procedure, a mixture of imine (0.005 mol) and 2-mercaptoacetic acid (0.006 mol) in benzene (20 mL) was refluxed with stirring for (12 hrs). The solvent was removed by using rotary evaporator. The remaining solid compound was neutralized by adding cold saturated sodium bicarbonate and allowed to stand overnight. The solid products were isolated by suction filtration, washed with water, dried, and then purified by flash chromatography (CHCl₃).

3a: R: H, C₂₃H₁₈N₂O₂S₂, mp. 232–234°C, yield: 70%. FT-IR (KBr cm⁻¹): 3021, 2930, 2845, 1667, 1606, 1510.

3b: R: 4-Cl, C₂₃H₁₇ClN₂O₂S₂, mp. 146–148°C, yield: 65%. FT-IR (KBr cm⁻¹): 3030, 2966, 2851, 1699, 1600, 1478; ¹H-NMR: 2.56: (s, 3H, CH₃); 3.91, 4.0 (dd, 2H, C₁₇); 6.19 (s, H, C₅); 7.6: (s, H, C₁₂); 8.08 (d, 2H, C_{8,8'}); 7.8 (d, H, C₁₅); 7.18–7.35 (m, 7H, for—C_{2,2'}; C_{3',3}; C_{7,7'}; C₁₄); 7.18–8.08 (m, 11H, for aromatic proton); ¹³C-NMR: 21.5: CH₃; 33.5: C₁₇; 64.53: C₅; 121.37: C₁₂; 122.7: C₁₅; 125.3: C_{7,7'}; 128.0: C₁₄; 128.18: C_{8,8'}; 128.3: C_{2,2}; 129.3: C_{3',3};

132.1: (C₉); 134.9: C₁; 135.2: C₁₃; 135.6: C₁₁; 137.5: C₄; 139.3: C₆; 152.18: C₁₆; 165.6: C₁₀; 170.9: C₁₈, (—C=O); Dept-135: 21.5: CH₃; 33.5: C₁₇; 64.53: C₅; 121.37: C₁₂; 122.7: C₁₅; 125.3: C_{7,7'}; 128.0: C₁₅; 128.18: C_{8,8'}; 128.3: C_{7,7'}; 129.3: C_{3',3};

3c: R: 3-Cl, C₂₃H₁₇ClN₂O₂S₂, mp. 195–197°C, yield: 70%. FT-IR (KBr cm⁻¹): 3032, 2925, 2850, 1683, 1600, 1595.

3d: R: 2-NO₂, C₂₃H₁₇N₃O₃S₂, mp. 210–212°C, yield: 69%. FT-IR (KBr cm⁻¹): 3032, 2920, 2840, 1699, 1600, 1521, 1501, 1320.

3e: R: 2-F, C₂₃H₁₇FN₂O₂S₂, mp. 198–200°C, yield: 72%. FT-IR (KBr cm⁻¹): 3027, 2916, 2835, 1681, 1607, 1511, 1485.

3f: R: 4-NO₂, C₂₃H₁₇N₃O₃S₂, mp. 230–231°C, yield: 75%. FT-IR (KBr): 3025, 2921, 2875, 1697, 1599, 1517, 1516, 1340; ¹H-NMR: 2.49 (s, 3H, CH₃); 3.97, 3.99 (dd, 2H, C₁₇); 6.35: (s, H, C₅); 6.69–8.5 (m, for all aromatic proton); ¹³C-NMR: 21.5: CH₃; 33.3: C₁₇; 63.9: C₅; 121.38: C_{2,2'}; 121.62: C₁₂; 122.8: C₁₅; 127.7: C_{7,7'}; 128.21: C₁₄; 128.5: C_{8,8'}; 129.6: C_{3,3'}; 132.4: C₉; 135.5: C₁₃; 135.1: C₁₁; 141.2: C₆; 146.2: C₆; 148.1: C₁; 152.31: C₁₆; 158.0: C₁₀; 170.6: C₁₈, (—C=O); Dept-135: 21.5: CH₃; 33.3: C₁₇; 63.9: C₅; 121.38: C_{2,2'}; 121.62: C₁₂; 122.8: C₁₅; 127.7: C_{7,7'}; 128.2: C₁₄; 128.5: C_{8',8}; 129.6: C_{3',3};

3g: R: 4-OCH₃, C₂₄H₂₀N₂O₂S₂, mp. 180–182°C, yield: 64%. FT-IR (KBr): 3020, 2995, 2917, 2845, 1668, 1590, 1510, 1253, 1024; ¹H-NMR: 2.56: (s, 3H, CH₃); 3.75 (s, 3H, -OCH₃); 3.9, 3.97 (dd, CH₂, C₁₇); 6.18: (s, H, C₁₂); 6.7 (d, 2H, C_{2,2'}); 6.8–8.4 (m, for aromatic ring); ¹³C-NMR: 21.56: CH₃; 33.68: C₁₇; 55.25: -OCH₃; 65.02: C₅; 114.34: C_{2,2'}; 121.38: C₁₂; 122.72: C₁₅; 125.61: C_{7,7'}; 128.01: C₁₄; 128.4: C_{8,8'}; 130.56: C₄; 130.8: C_{3',3}; 131.93: C₉; 135.26: C₁₃; 135.52: C₁₁; 139.65: C₆; 152.2: C₁₆; 160.04: C₁; 165: C₁₀; 171: C₁₈; Dept-135: 21.56: CH₃; 33.68: C₁₇; 55.25: -OCH₃; 65.02: C₅; 114.34: C_{2,2'}; 121.31: C₁₂; 122.73: C₁₅; 125.61: C_{7,7'}; 128.01: C₁₄; 128.4: C_{8,8'}; 130.8: C_{3',3}.

3j: R: 3-NO₂, C₂₃H₁₇N₃O₃S₂, mp. 180–182°C, yield: 70%. FT-IR (KBr cm⁻¹): 3032, 2917, 2860, 1682, 1600, 1519, 1486, 1481, 1369.

4-(6-methylbenzo[d]thiazol-2-yl)benzenamine (1) was treated with different substituted benzaldehydes to produce a series of Schiff base compounds (2a-j). The Schiff bases were subjected to cyclocondensation reactions with 2-mercaptoacetic acid in benzene to produce a series of new thiazolidin-4-one derivatives (3a-j), Scheme 1.

In the study of the influence of the substituents of the substituted benzaldehydes on the rate of the formation and yield of imine products, any factor increasing the electrophilicity of the carbonyl carbon of benzaldehyde should increase reactivity. In contrast, substituents reducing electrophilicity should reduce reactivity. In contrast to electron-withdrawing substituents the electron-releasing substituents, release electrons to the ring and disperse the positive charge on carbonyl carbon, as a result the rate and the yield of imine reduce.

TABLE 1: Antibacterial activity of some prepared imines and thiazolidin-4-ones with inhibition zone diameters in (mm) scale against *S.aureus* and *E.coli* bioorganisms.

Product no.	<i>E. coli</i> G (–ve)	<i>S.aureus</i> G (+ve)
TMP	40	30
2f	40	30
2g	35	30
3f	40	30
3b	35	30
3h	40	35
3g	38	30

The chemical structures of the synthesized compounds were confirmed by IR, ^1H -NMR and ^{13}C -NMR, and Dept-135. The FT-IR spectra of the synthesized imines (2a–j) showed the disappearance of amine and carbonyl bands and appearance of C=N group band around 1620 cm^{-1} considered as a good evidence for the formation of imine groups.

The ^1H -NMR spectra of all imines, (Figure 1) show a singlet signals at 8.4 ppm [21], belongs to imine or azomethene (CH=N–) group, with a characteristic bands for each compound such as compound (2b) which has showed a singlet at (2.5 ppm) due to three protons of CH_3 group, compound (2g) showed a singlet at 3.9 ppm which belongs to three protons of OCH_3 group, and compound (2i) showed a singlet at 10.1 for CHO group. The ^{13}C -NMR and Dept spectra (Figures 2 and 3) for each compound were matching perfectly with the expectations. The IR spectra of thiazolidin-4-ones showed a characteristic band at $(1667\text{--}1699)\text{ cm}^{-1}$ due to carbonyl group stretching of cyclic amides [22]. The disappearance of imine bands at 1620 cm^{-1} indicates the formation of thiazolidin-4-one ring. The ^1H -NMR spectra of thiazolidin-4-ones, (Figure 4), showed characteristic signals dd at (3.8, 4) and a singlet at 6 ppm corresponding to proton of C_{17} and C_5 of thiazolidin-4-ones ring, confirming the nonequivalence of protons at C_{17} of the five-membered ring of thiazolidin-4-ones, which appeared as two doublet to doublet (dd) and a singlet of monoproton of (C_5). Also as imines the ^{13}C -NMR and Dept spectra (Figures 5 and 6) for each compound were matching perfectly with the expectations, and the Dept spectra for thiazolidin-4-ones showed a downward signal [23] for diprotonated carbon (CH_2) group at -65 ppm of the synthesized thiazolidin-4-one rings.

Antibacterial Activity. The antimicrobial activity of the synthesized compounds were screened against two types of bacteria *Escherichia coli* (gram negative) and *Staphylococcus aureus* (gram positive) by using the cup-plate agar diffusion method with KBr disc of compounds. The prepared discs were placed on the surface of the cultured media with each of the two bacteria incubated for 24 hours at 37°C , and the results were monitored by measuring inhibition zones in mm. The screening results are listed in (Table 1) and they show that most prepared compounds were sensitive against both types of bacteria. The antibacterial activity of these compounds

shows similar activity with (TMP) trimethoprim antibiotic, which prevents infections of the urinary tract caused by bacteria.

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