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Microwave-assisted synthesis and biological activity of new Schiff bases derived from dimers of 4-amino-3-[3-(1-benzyl)indole]-5-thiomethyl-1,2,4-triazole

Yongle Peng · Zhigang Zhao · Xingli Liu · Guohua Li

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Abstract An expeditious method with microwave irradiation has been developed for synthesis of novel Schiff bases from dimers of 4-amino-3-[3-(1-benzyl)indole]-5-thiomethyl-1,2,4-triazole. Its distinct advantages are short reaction times and good conversion. The structures of these new Schiff bases were established by ^{1}H NMR, IR, and mass spectroscopy, and elemental analysis. The antibacterial activity of ten novel Schiff bases against three bacterial strains was studied by the disk diffusion method. Preliminary results indicated that some of the compounds had strong antibacterial activity.

Keywords Microwave irradiation · Synthesis · Indole · Schiff base · 1,2,4-Triazole

Introduction

During the last few years derivatives of indole have attracted much attention because of their special biological activity in medicine and agriculture. Andreani et al. [1–6] reported that indole derivatives have substantial biological activity, for example cytotoxic, antibacterial, antiviral, anticancer, and antiproliferative, and as plant growth regulators. Furthermore, the triazole structure is occurs widely in natural medicines and synthetic drugs. Currently available anti-microbial [7] or anti-tumor [8] drugs, for example terconazole, itraconazole, fluconazole, fosfluconazole, vibunazole, letrozole, anastrozole, and vorozole all contain the triazole ring. Moreover, Schiff base compounds [9–13] and heterocyclic sulfide compounds [14] were also reported to have cytotoxic, anticonvulsant, antiproliferative, anticancer,

Y. Peng · Z. Zhao (✉) · X. Liu · G. Li
College of Chemistry and Environmental Protection Engineering, Southwest University for Nationalities, Chengdu 610041, China
e-mail: zzg63129@yahoo.com.cn

antifungal, and antibacterial activity. In view of these research results and because of the principle of active superposition, we synthesized a series of triazole Schiff bases containing indole and heterocyclic sulfide structures. We can expected this kind of new triazole Schiff base to have good biological activity. We also hoped they could be used for other applications in the chemical industry.

Microwave synthesis, because of its unique advantages, has been widely applied in organic synthesis in recent years [15–17]. We are interested in the pharmaceutical synthesis using microwave methods [18–21]. As a continuation of this work, herein we report the synthesis, under microwave irradiation, of novel triazole Schiff bases containing indole and heterocyclic sulfide structures. The synthetic route is shown in Scheme 1.

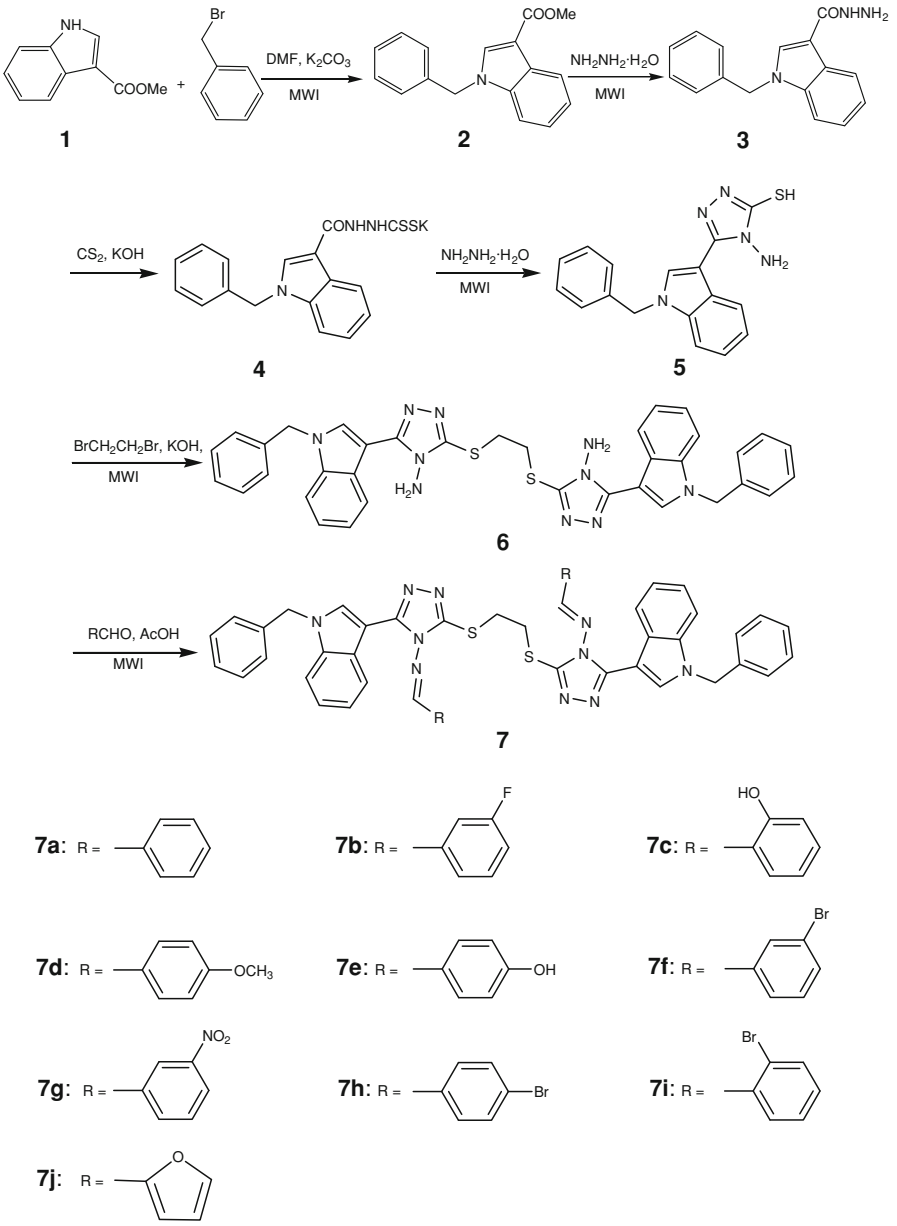
Results and discussion

Spectroscopic studies

The structures of **6** and **7a–j** were confirmed on the basis of spectral data and elemental analysis. The IR spectrum of **6** contained an absorption band at 3,326–3,123 cm^{-1}, because of the NH_2 group; this was absent from the IR spectra of **7a–j**. Strong bands at 1,588–1,526 cm^{-1} were assigned to absorption of C=N. The ^1H NMR spectrum of **6** contained a singlet peak at 6.16 ppm because of the NH_2 group; this was absent from the spectra of compounds **7a–j**. The singlet peak at 9.10–8.45 ppm was assigned to ArCHN. The protons of ArH appeared at 8.69–6.91 ppm. The singlet peak at approximately 5.45 ppm was assigned to $ArCH_2N$. The singlet peak at approximately 3.53 ppm was assigned to SCH_2CH_2S. In the ^{13}C NMR spectra of compounds **6** and **7a–j**, the peak at approximately 50 ppm was assigned to $PhCH_2N$, the carbon atom of SCH_2CH_2S appeared at approximately 33 ppm. Other peaks were assigned to carbon atoms of the aromatic nucleus and the indole ring. Their ESI mass spectra contained the correct molecular ions with high intensity.

Antibacterial studies

The newly synthesized compounds were screened for their antibacterial activity against *Escherichia coli* (ATCC-25922), *Staphylococcus aureus* (ATCC-25923), and *Pseudomonas aeruginosa* (ATCC-27853) bacterial strains by the disk-diffusion method [22, 23]. A standard inoculum, prepared from beef extract, peptone, sodium chloride, blood serum, and agar, was applied to the surface of sterile agar plates. A sterile glass spreader was used for even distribution of the inoculum. Disks 6.25 mm in diameter were prepared from ordinary filter paper and sterilized by dry heat at 140 °C for an hour. The sterile disks, previously soaked in a fixed concentration (500 μg/mL) of the test compounds, were placed on the nutrient agar medium. The plates were inverted and incubated at 37 °C for 24 h. Solvent and growth control experiments were also conducted. Sparfloxacin was used as standard drug. The results from testing of bacterial inhibition are list in Table 1.



Scheme 1 Synthetic route for new Schiff bases derived from dimers of 4-amino-3-[3-(1-benzyl)indole]-5-thiomethyl-1,2,4-triazole

As is obvious from Table 1, among the compounds screened, compounds **7b**, **7d**, and **7f** are the most biologically effective. Compound **7f** had good antibacterial activity against all three bacteria, compound **7b** had satisfactory activity against *E. coli* and *P. aeruginosa*, and compound **7d** strongly inhibited growth of *S. aureus*.

Table 1 Bacterial inhibition results for ten novel triazole Schiff bases

Compound		Bacteria		
		Escherichia coli (ATCC-25922)	*Staphylococcus aureus* (ATCC-25923)	*Pseudomonas aeruginosa* (ATCC-27853)
7a	R=C_6H_5	−	−	−
7b	R=3-FC_6H_4	++	+	++
7c	R=2-HOC_6H_4	−	−	−
7d	R=4-$CH_3OC_6H_4$	+	++	−
7e	R=4-HOC_6H_4	−	−	+
7f	R=3-BrC_6H_4	++	++	++
7g	R=2-$O_2NC_6H_4$	−	−	+
7h	R=4-BrC_6H_4	+	+	−
7i	R=2-BrC_6H_4	+	+	−
7j	R=2-furyl	−	−	−
Sparfloxacin		++	++	++

++, strong activity; +, moderate activity; −, weak or poor activity

So, we have reason to believe these three compounds or their derivatives can be developed as antibacterial drugs.

Comparison of microwave irradiation and conventional heating

As shown in Table 2, microwave irradiation had two large advantages over conventional heating—reaction times were reduced from 1,440–1,800 min to 4–8 min, i.e. reaction was 225–360 times faster, and yields increased from 72–78 % to 85–94 %. We can therefore conclude that the microwave-enhanced procedure enables rapid and efficient synthesis of triazole Schiff base compounds containing indole and heterocyclic sulfide structures.

Experimental

Melting points were measured on a micro-melting point apparatus and are uncorrected. Infrared spectra were obtained with a 1700 Perkin–Elmer FTIR using KBr disks. NMR spectra were recorded on a Varian Inova 400-MHz spectrometer with TMS as internal standard, DMSO-d_6 or pyridine-d_5 as solvent, operating at 400 MHz for ^{1}H and 100 MHz for ^{13}C. Mass spectra were determined on Finnigan LCQ^{DECA} instrument. Elemental analysis was performed with a Carlo Erba 1106 autoanalyzer. All microwave-assisted reactions were performed in a commercial microwave reactor (XH-100A, 100–1,000 W; Beijing Xianghu Science and Technology Development, Beijing, PR China). All solvents were purified before use. Intermediates **2**, **3**, **4**, and **5** were prepared in accordance with reported procedures [24, 25].

Table 2 Comparison of microwave irradiation and conventional heating for synthesis of triazole Schiff base compounds **7a–j**

Compound		Traditional method		Microwave method		t_c/t_m[a]
		Time (min)	Yield (%)	Time (min)	Yield (%)	
7a	R=C_6H_5	1,440	75	4	86	360
7b	R=3-FC_6H_4	1,560	75	5	85	312
7c	R=2-HOC_6H_4	1,680	76	6	87	280
7d	R=4-$CH_3OC_6H_4$	1,440	70	5	86	288
7e	R=4-HOC_6H_4	1,500	78	5	91	300
7f	R=3-BrC_6H_4	1,620	76	5	86	324
7g	R=2-$O_2NC_6H_4$	1,800	72	8	90	225
7h	R=4-BrC_6H_4	1,500	78	6	87	250
7i	R=2-BrC_6H_4	1,680	76	7	94	240
7j	R=2-furyl	1,800	74	5	88	360

[a] t_c, time required by conventional method; t_m, time required by microwave method

Procedure for preparation of intermediate **6**

Compound **5** (1.7 mmol), KOH (1.6 mmol), 1,2-dibromoethane (0.8 mmol), and methanol (5 mL) were placed in a vessel which was then sealed and shaken to homogenize the mixture. The mixture was irradiated at 350 W for 10 min. After cooling to room temperature, filtration, and recrystallization, we obtained the pure compound **6** as a white solid, yield 91 %, m.p. 247–249 °C; IR (KBr) (cm^{-1}): 3326, 3123, 1622, 1581, 1448, 1386, 1338, 1187, 1021, 930, 740; ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 8.50 (s, 2H, ArH), 8.31(d, $J = 7.6$ Hz, 2H, ArH), 7.55(d, $J = 8.0$ Hz, 2H, ArH), 7.33–7.15 (m, 14H, ArH and indole-CH); 6.16(s, 4H, NH_2), 5.46 (s, 4H, $PhCH_2N$), 3.34 (s, 4H, SCH_2CH_2S); ^{13}C NMR (100 MHz, DMSO-d_6, ppm) δ: 152.08, 151.10, 138.14, 136.06, 129.71, 129.06, 127.98, 127.53, 126.45, 122.90, 122.24, 121.03, 111.00, 102.08, 49.91, 31.91; ESI–MS *m/z* (%): 669 [$(M + 1)^+$, 100]. Anal. Calcd. for $C_{36}H_{32}N_{10}S_2$: C, 64.65; H, 4.82, N, 20.94. Found: C, 64.70; H, 4.75; N, 20.87 %.

Conventional method for preparation of compounds **7a–j**

Compound **6** (0.12 mmol), aldehyde (0.27 mmol), and glacial acetic acid (5 mL) were placed in a round-bottomed flask and the reaction mixture was heated to reflux for 24–30 h. When the reaction was complete (monitored by TLC) the flask was cooled to room temperature and evaporated to remove the glacial acetic acid. Ethanol (5 ml) was added into the residue. Solid formed immediately and was collected by filtration and recrystallized from *N*,*N*-dimethylformamide to furnish the pure compounds **7a–j**.

Microwave method for preparation of compounds **7a–j**

Compound **6** (0.12 mmol), acetic acid (2 mL), and aldehyde (0.27 mmol) were mixed thoroughly in a sealed vessel. The vessel was then placed in a microwave oven and the mixture was irradiated at 300 W for 4–8 min. The reaction was monitored by TLC until it was complete. The acetic acid was then removed by reduced-pressure distillation. Ethanol (10 mL) was added to the residue. The solid formed was isolated by filtration. After recrystallization, the pure product **7a–j** was obtained as a white, yellow, or green solid. The physical and spectra data of compounds **7a–j** are as follows.

7a: Yellow solid, yield 86 %, m.p. 230–232 °C; IR (KBr)(cm^{-1}): 3055, 1578, 1439, 1387, 747, 693; ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 8.88 (s, 2H, ArCH=N), 8.27 (d, J = 7.6 Hz, 2H, ArH), 7.90–7.87 (m, 6H, ArH), 7.66–7.52 (m, 8H, ArH), 7.26–7.16 (m, 14H, ArH and indole-CH), 5.44 (s, 4H, $PhCH_2N$), 3.53 (s, 4H, SCH_2CH_2S); ^{13}C NMR (100 MHz, Pyridine-d_5, ppm) δ: 165.48, 144.49, 137.54, 137.02, 136.12, 135.09, 133.31, 132.54, 129.96, 129.70, 129.51, 129.24, 128.23, 127.78, 127.23, 123.06, 121.71, 110.80, 103.20, 50.26, 33.80; ESI–MS m/z (%): 845 [$(M + 1)^+$, 100]. Anal. Calcd. for $C_{50}H_{40}N_{10}S_2$: C, 71.07; H, 4.77, N, 16.58. Found: C, 71.13; H, 4.70; N, 16.54 %.

7b: Pale yellow solid, yield 85 %, m.p. 232–234 °C; IR (KBr)(cm^{-1}): 3063, 1615, 1583, 1449, 1379, 1270, 742; ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 8.91 (s, 2H, ArCH=N), 8.25 (d, 2H, ArH), 7.90 (s, 2H, ArH), 7.74–7.50 (m, 10H, ArH), 7.26–7.15 (m, 14H, ArH and indole-CH), 5.44 (s, 4H, $PhCH_2N$), 3.53 (s, 4H, SCH_2CH_2S); ^{13}C NMR (100 MHz, Pyridine-d_5, ppm) δ: 164.42, 163.97, 161.97, 144.44, 137.38, 136.88, 134.62, 131.50, 131.42, 129.89, 129.10, 128.13, 127.69, 127.14, 127.02, 125.88, 122.96, 121.60, 120.13, 119.91, 115.24, 115.01, 110.77, 102.85, 50.20, 33.81; ESI–MS m/z (%): 881 [$(M + 1)^+$, 100]. Anal. Calcd. for $C_{50}H_{38}F_2N_{10}S_2$: C, 68.16; H, 4.35; N, 15.90. Found: C, 68.21; H, 4.29; N, 15.87 %.

7c: Pale yellow solid, yield 87 %, m.p. 209–211 °C; IR (KBr)(cm^{-1}): 3242, 3023, 1587, 1442, 1371, 1164, 737, 693; ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 10.56 (s, 2H, ArOH), 9.00 (s, 2H, ArCH=N), 8.28 (d, J = 7.6 Hz, 2H, ArH), 7.96(s, 2H, ArH), 7.88(d, J = 7.6 Hz, 2H, ArH), 7.59 (d, J = 8.0 Hz, 2H, ArH), 7.46 (t, J = 8.4 Hz, 2H, ArH), 7.24–7.16 (m, 14H, ArH and indole-CH), 7.00–6.92 (m, 4H, ArH), 5.46 (s, 4H, $PhCH_2N$), 3.56 (s, 4H, SCH_2CH_2S); ^{13}C NMR (100 MHz, Pyridine-d_5, ppm) δ: 162.13, 158.91, 143.22, 136.08, 135.47, 133.60, 128.48, 127.73, 127.70, 126.68, 126.22, 125.82, 125.82, 125.80, 121.98, 121.73, 120.14, 118.80, 117.86,116.06, 109.38, 101.83, 48.95, 32.36; ESI–MS m/z (%): 877 [$(M + 1)^+$, 100]. Anal. Calcd. for $C_{50}H_{40}N_{10}O_2S_2$: C, 68.47; H, 4.60; N, 15.97. Found: C, 68.53; H, 4.53; N, 15.95 %.

7d: White solid, yield 86 %, m.p. 210–212 °C; IR (KBr)(cm^{-1}): 3038, 1588, 1427, 1249, 1161, 1023, 752; ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 8.71 (s, 2H, ArCH=N), 8.28 (d, J = 7.6 Hz, 2H, ArH), 7.85 (t, J = 8.8 Hz, 6H, ArH), 7.59 (d, J = 8.0 Hz, 2H, ArH), 7.24–7.18 (m, 14H, ArH and indole-CH), 7.08 (d, J = 8.4 Hz, 4H, ArH), 5.43 (s, 4H, $PhCH_2N$), 3.85 (s, 6H, OCH_3), 3.51 (s, 4H, SCH_2CH_2S); ^{13}C NMR (100 MHz, Pyridine-d_5, ppm) δ: 171.43, 164.38, 147.92, 136.09, 135.50, 130.07, 128.38, 127.73, 126.67, 126.19, 125.74, 123.57, 122.58,

121.85, 121.85, 121.55, 120.16, 113.77, 109.37, 101.85, 54.16, 48.87, 32.40; ESI–MS *m/z* (%): 905 [(M + 1)$^+$, 100]. Anal. Calcd. for $C_{52}H_{44}N_{10}O_2S_2$: C, 69.00; H, 4.90; N, 15.48. Found: C, 69.09; H, 4.84; N, 15.45 %.

7e: White solid, yield 91 %, m.p. 269–271 °C; IR (KBr)(cm^{-1}): 3233, 3023, 1587, 1442, 1371, 1265, 1164, 737; ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 10.50 (s, 2H, PhOH), 8.70 (s, 2H, ArCH=N), 8.27 (d, *J*=7.6 Hz, 2H, ArH), 7.85–7.74 (m, 6H, ArH), 7.57 (d, $J = 8.0$ Hz, 2H, ArH), 7.26–7.16 (m, 14H, ArH and indole-CH), 6.91 (d, $J = 8.4$ Hz, 4H, ArH), 5.46 (s, 4H, $PhCH_2N$), 3.32 (s, 4H, SCH_2CH_2S); ^{13}C NMR (100 MHz, Pyridine-d_5, ppm) δ: 165.05, 162.55, 148.19, 143.18, 136.08, 135.47, 130.58, 128.35, 127.70, 126.64, 126.20, 125.77, 122.03, 121.86, 121.77, 120.12, 115.80, 109.34, 101.92, 48.88, 32.37; ESI–MS *m/z* (%): 877[(M + 1)$^+$, 100]. Anal. Calcd. for $C_{50}H_{40}N_{10}O_2S_2$: C, 68.47; H, 4.60; N, 15.97. Found: C, 68.55; H, 4.58; N,15.94 %.

7f: Pale white solid, yield 86 %, m.p. 216–218 °C; IR (KBr)(cm^{-1}): 3054, 2924, 1585, 1437, 1372, 1168, 740; ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 8.89 (s, 2H, ArCH=N), 8.24 (d, $J = 8.0$ Hz, 2H, ArH), 8.09 (s, 2H, ArH), 7.89–7.82 (m, 6H, ArH), 7.60–7.46 (m, 4H, ArH), 7.29–7.15 (m, 14H, ArH), 5.43 (s, 4H, $PhCH_2N$), 3.53 (s, 4H, SCH_2CH_2S); ^{13}C NMR (100 MHz, Pyridine-d_5, ppm) δ: 161.88, 143.06, 136.06, 135.51, 133.20, 130.45, 129.89, 128.55, 127.77, 127.70, 126.76, 126.66, 126.30, 125.78, 125.67, 121.62, 120.24, 109.40, 101.51, 48.87, 32.45; ESI–MS *m/z* (%): 1003 [(M + 1)$^+$, 100]. Anal. Calcd. for $C_{50}H_{38}Br_2N_{10}S_2$: C, 59.88; H, 3.82; N,13.97. Found: C, 59.91; H,3.76; N, 13.95 %.

7g: Yellow solid, yield 90 %, m.p. 231–233 °C; IR (KBr)(cm^{-1}): 3071, 2365, 1578, 1526, 1439, 1347, 738, 688; ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 9.09 (s, 2H, ArCH=N), 8.69 (s, 2H, ArH), 8.43 (d, $J = 8.4$ Hz, 2H, ArH), 8.31–8.23 (m, 4H, ArH), 7.93 (s, 2H, ArH), 7.80 (t, $J = 8.0$ Hz, 2H, ArH), 7.59 (d, $J = 8.0$ Hz, 2H, ArH), 7.21–7.15 (m, 14H, ArH and indole-CH), 5.44 (s, 4H, $PhCH_2N$), 3.56 (s, 4H, SCH_2CH_2S); ^{13}C NMR (100 MHz, Pyridine-d_5, ppm) δ: 161.34, 144.62, 138.10, 137.63, 135.07, 134.47, 133.70, 130.87, 130.23, 130.11, 129.28, 128.26, 128.06, 127.79, 127.29, 127.14, 126.89, 124.60, 123.02, 121.83, 116.85, 50.11, 32.33; ESI–MS *m/z* (%): 935 [(M + 1)$^+$, 100]. Anal. Calcd. for $C_{50}H_{38}N_{12}O_4S_2$: C, 64.23; H, 4.10; N, 17.98. Found: C, 64.29; H, 4.02; N, 17.96 %.

7h: Pale green solid, yield 87 %, m.p. 235–237 °C; IR (KBr)(cm^{-1}): 3036, 1576, 1423, 1390, 1299, 1005, 750; ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 8.87 (s, 2H, ArCH=N), 8.25 (d, $J = 8.0$ Hz, 2H, ArH), 7.87 (s, 2H, ArH), 7.81–7.71 (m, 8H, ArH), 7.58 (d, $J = 8.4$ Hz, 2H, ArH), 7.26–7.16 (m, 14H, ArH and indole-CH), 5.48 (s, 4H, $PhCH_2N$), 3.52 (s, 4H, SCH_2CH_2S); ^{13}C NMR (100 MHz, DMSO-d_6, ppm) δ: 166.42, 148.52, 143.56, 137.10, 135.68, 132.27, 130.67, 128.46, 127.48, 127.26, 126.95, 125.53, 122.58, 121.41, 120.74, 115.80, 110.62, 100.95, 49.18, 29.46; ESI–MS *m/z* (%): 1003 [(M + 1)$^+$, 100]. Anal. Calcd. for $C_{50}H_{38}Br_2N_{10}S_2$: C, 59.88; H, 3.82; N 13.97. Found: C 59.92, H 3.73, N 13.94 %.

7i: Pale white solid, yield 94 %, m.p. 240–242 °C; IR (KBr)(cm^{-1}): 3058, 2925, 1580, 1442, 1419, 1202, 1169, 1026, 745; ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 9.10 (s, 2H, ArCH=N), 8.18(d, $J = 8.4$ Hz, 2H, ArH), 8.03–8.00 (m, 4H, ArH), 7.80 (t, $J = 8.0$ Hz, 2H, ArH), 7.61–7.47 (m, 6H, ArH), 7.28–7.15 (m, 14H, ArH and indole-CH), 5.49 (s, 4H, $PhCH_2N$), 3.60 (s, 4H, SCH_2CH_2S); ^{13}C NMR (100 MHz,

Pyridine-d_5, ppm) δ: 160.13, 143.28, 136.55, 136.17, 135.61, 133.56, 132.74, 132.58, 130.16, 128.80, 128.04, 127.09, 126.72, 126.03, 125.78, 125.63, 120.20, 109.44, 101.58, 48.84, 32.41; ESI–MS *m/z* (%): 1003 [$(M + 1)^+$, 100]. Anal. Calcd. for $C_{50}H_{38}Br_2N_{10}S_2$: C, 59.88; H, 3.82; N,13.97. found: C, 59.94; H, 3.78; N; 13.95 %.

7j: Yellow solid, yield 88 %, m.p. 214–216 °C; IR (KBr)(cm^{-1}): 3031, 1583, 1442, 1386, 1291, 1156, 736, 702; ^{1}H NMR (400 MHz, DMSO-d_6, ppm) δ: 8.76 (s, 2H, ArCH=N), 8.25 (d, $J = 7.6$ Hz, 2H, ArH), 8.11 (s, 2H, ArH,), 7.91 (s, 2H, ArH), 7.57 (d, $J = 8.0$ Hz, 2H, 5-furyl-H), 7.34 (d, $J = 3.6$ Hz, 2H, 3-furyl-H), 7.29–7.16 (m, 14H, ArH), 6.80–6.79 (m, 2H, 4-furyl-H), 5.47 (s, 4H, $PhCH_2N$), 3.52 (s, 4H, SCH_2CH_2S); ^{13}C NMR (100 MHz, Pyridine-d_5, ppm) δ: 153.34, 148.40, 148.26, 144.64, 137.60, 136.95, 130.24, 129.52, 129.22, 128.15, 127.64, 127.29, 123.06, 121.69, 120.46, 113.45, 110.91, 103.06, 50.46, 33.95; ESI–MS *m/z* (%): 825 [$(M + 1)^+$, 100]. Anal. Calcd. for $C_{46}H_{36}N_{10}O_2S_2$: C, 66.97; H, 4.40; N, 16.98. Found: C, 67.90; H, 4.38; N,16.94 %.

Conclusions

We have developed a facile and efficient method for synthesis, under microwave irradiation, of new Schiff bases derived from dimers of 4-amino-3-[3-(1-benzyl)indole]-5-thiomethyl-1,2,4-triazole. The reactions proceeded much more quickly and yields were higher than when conventional heating was used. Many of the newly synthesized compounds had good antibacterial activity, especially those containing methoxyphenyl and halogen substituents. We therefore believe there is ample scope for further study and our work may have a beneficial outcome.

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