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ORIGINAL ARTICLE

Synthesis, structure analysis and antibacterial activity of *N*-[5-dehydroabietyl-[1,3,4]thiadiazol-2-yl]-aromatic amide derivatives

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KEYWORDS

N-[5-Dehydroabietyl-[1,3,4]thiadiazol-2-yl]-aromatic amide;
Dehydroabietic acid;
Crystal structure;
Minimum inhibition concentration;
Antibacterial activity

Abstract A series of *N*-[5-dehydroabietyl-[1,3,4]thiadiazol-2-yl]-aromatic amide derivatives were synthesized from dehydroabietic acid. Their structures were characterized by IR, ^{1}H NMR, MS, elemental analysis and single crystal X-ray diffraction. Four six-membered rings of 2b exhibited plane, half-chair and chair configurations, respectively. The antibacterial activity of these newly synthesized *N*-[5-dehydroabietyl-[1,3,4]thiadiazol-2-yl]-aromatic amides derivatives against Gram-negative bacteria and Gram-positive bacteria was also investigated using the minimum inhibition concentration method. Preliminary results indicated that five compounds displayed better antibacterial activity against *Pseudomonas aeruginosa* and *Escherichia aerogenes* compared to commercially available antibacterial agents of Bromogeramine and Ampicillin sodium.

1. Introduction

Dehydroabietic acid (DHA) is a naturally occurring diterpenic resin acid, which can be easily isolated from commercial disproportionated rosin by crystallization of the 2-aminoethanol salt. DHA can be used in the field of synthetic chemistry because of its unique characteristics. DHA and a number of its derivatives have been reported to possess a broad spectrum of biological properties such as antimicrobial, antitumor, antiviral, antioxidant, gastroprotective and K+ channel-opening activities [1–6].

Amides are important organic compounds with a wide range of applications [7,8]. The stablility of amide bond makes the amide function important to synthetic chemists especially in peptides and lactam synthesis, in which the formation of amide bonds is crucial. Some derivatives of amides exhibit

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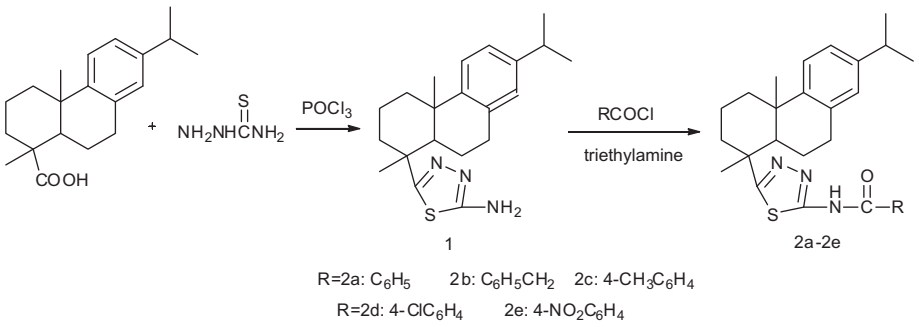

Scheme 1 Synthetic route of target compounds.

biological properties such as anticancer, antihistamine, antifungal, and antibacterial [9–13].

The synthesis of compounds incorporated with a 1,3,4-thiadiazole ring has attracted widespread attention due to its diverse pharmacological properties such as antimicrobial [14,15], antifungal [16], anti-inflammatory [17], antitubercular [18], herbicidal [19,20] and plant growth regulating activities [21–24]. Although there are a number of antibiotics which are commercially used in medicine, the synthesis of new compounds is of vital importance due to the increase of drug resistance. Moreover, it is important to obtain compounds with higher bioactivity and lower toxic effect.

In this paper, a series of *N*-[5-dehydroabietyl-[1,3,4] thiadiazol-2-yl]-aromatic amide derivatives were synthesized from dehydroabietic acid with the aim of finding potent bioactive compounds. The structures of these compounds were verified by means of IR, MS, ^{1}H NMR and elemental analysis. In addition, the preliminary antimicrobial activity was tested. The synthetic route of target compounds is shown in Scheme 1.

2. Experiment

2.1. General methods and materials

Dehydroabietic acid was separated and purified from disproportionated rosin. All other chemicals were of reagent grade.

The IR spectra were taken on a Nicolet IS10 FT-IR (Nicolet, Madison, USA) spectrophotometer. The ^{1}H NMR spectra were recorded on a Bruker AV-300 (Bruker, Karlsruhe, Germany) nuclear magnetic resonance spectrometer with DMSO as solvent and TMS as internal standard. The MS spectra were taken on an Agilent-5973 (Agilent, Santa Clara, USA) spectrophotometer. The melting points were determined using WRS-2(Shanghai precision & scientific instrument Co., LTD, China) melting point apparatus. The elemental analysis (C, H, N) was performed on a Vario EL-III (Elementar, Hanau, Germany) elemental analyzer, their results were found to be in good agreement with the calculated values (within ±0.8%). All reactions were traced by Thin layer chromatography (TLC).

*2.2. General synthetic procedure for 5-dehydroabietyl-2-azyl-1,3,4-thiadiazole (**1**)*

Dehydroabietic acid (0.05 mol) was refluxed with thiosemicarbazide (0.05 mol) in the presence of phosphorus oxychloride (15 ml) for 1 h. The reaction mixture was cooled and diluted with water and again refluxed for 4 h. The reaction was monitored by thin layer chromatography and filtered after completion. The filtrate was basified with potassium hydroxide and the precipitate was filtered off and then recrystallized from ethanol to give the desired compound **1**. Gray white solid. Yield: 72.6%. Mp: 228.6–229.1 °C. IR: = 3293, 3124, 2935, 2870, 1650,1497, 1459, 670 cm^{-1}. MS(ESI (+)) *m*/*z*: 356 (M + H$^+$), 378 (M + Na$^+$). ^{1}H NMR (DMSO, 300 MHz): δ = 9.21 (s, 2H, –NH$_2$–) 6.85–7.21 (m, 3H, Ar–H), 2.70–2.85 (m, 2H, –CH–), 1.72–2.61 (m, 10H, –CH$_2$–), 1.14–1.67 (m, 12H, –CH$_3$). Calc. for $C_{21}H_{29}N_3S$, %: C, 70.94; H, 8.22; N, 11.82. Found, %: C, 70.50; H, 8.29; N, 11.52.

*2.3. General synthetic procedure for N-[5-dehydroabietyl-[1,3,4]thiadiazol-2-yl]-aromatic amide (**2a**–**2e**)*

A solution of aryl chloride in 15 mL CH_2Cl_2 was added dropwise to a solution of 10 mmol of compound **1** and 30 mmol triethylamine in 40 ml CH_2Cl_2 within 30 min at temperature 0–5 °C. The reaction mixture was then allowed to warm to room temperature over 6 h and washed with water, then dried with anhydrous $MgSO_4$. The residue was purified using silica gel chromatography [*v*(ethyl acetate)/*v*(petroleum ether) = 10:1) to give compounds **2a**–**2e**.

*2.3.1. N-[5-dehydroabietyl-[1,3,4]thiadiazol-2-yl]-benzamide(**2a** $C_{28}H_{33}N_3OS$)*

White solid. Yield: 85%. Mp: 184.6–185.5 °C. IR: = 3149, 2955, 2866, 1667, 1601, 1581, 1494, 1451, 676 cm^{-1}. MS(ESI (+)) *m*/*z*: 460 (M + H$^+$), 482 (M + Na$^+$). ^{1}H NMR (DMSO, 300 MHz): δ = 12.91(s, H, –CONH–), 7.49–8.08 (m, 5H, Ar–H), 6.80–7.18 (m, 3H, Ar–H), 2.71–2.80 (m, 2H, –CH–), 2.02–2.69 (m, 10H, –CH$_2$–), 1.41–1.98 (m, 12H, –CH$_3$). Calc. for $C_{28}H_{33}N_3OS$, %: C, 73.16; H, 7.24; N, 9.14. Found, %: C, 73.30; H, 7.58; N, 9.37.

*2.3.2. N-[5-dehydroabietyl-[1,3,4]thiadiazol-2-yl]-phenylacetamide (**2b** $C_{29}H_{35}N_3OS$)*

White solid. Yield: 80.5%. Mp: 161.8–162.3 °C. IR: = 3152, 2922, 2859, 1694, 1603, 1495, 1453, 653 cm^{-1}. MS(ESI (+)) *m*/*z*: 474 (M + H$^+$), 496 (M + Na$^+$). ^{1}H NMR (DMSO, 300 MHz): δ = 12.63 (s, H, –CONH–), 7.14–7.30 (m, 5H, Ar–H), 6.78–6.96 (m, 3H, Ar–H), 3.76 (s, 2H, –NHCO–CH$_2$–), 2.69–2.77 (m, 2H, –CH–), 2.30–2.63 (m, 10H, –CH$_2$–), 1.37–1.95 (m, 12H, –CH$_3$). Calc. for $C_{29}H_{35}N_3OS$, %: C, 73.53; H, 7.45; N, 8.87. Found, %: C, 73.93; H, 7.83; N, 9.04.

2.3.3. N-[5-dehydroabietyl-[1,3,4]thiadiazol-2-yl]-p-methyl benzamide (**2c** $C_{29}H_{35}N_3OS$)

White solid. Yield: 83.2%. Mp: 183.1–184.0 °C. IR: = 3150, 2928, 2869, 1660, 1609, 1508, 1463, 681 cm^{-1}. MS(ESI (+)) *m/z*: 474 ($M+H^+$), 496 ($M+Na^+$). ^1H NMR (DMSO, 300 MHz): δ = 12.79 (s, H, –CONH–), 7.16–7.99 (m, 4H, Ar–H), 6.81–6.98 (m, 3H, Ar–H), 2.70–2.81(m, 3H, Ar–CH_3), 2.36–2.67 (m, 2H, –CH–), 1.71–2.02 (m, 10H, –CH_2–), 1.30–1.52 (m, 12H, –CH_3). Calc. for $C_{29}H_{35}N_3OS$, %: C, 73.53; H, 7.45; N, 8.87. Found, %: C, 73.76; H, 7.80; N, 8.89.

2.3.4. N-[5-dehydroabietyl-[1,3,4]thiadiazol-2-yl]-p-chloro benzamide (**2d** $C_{28}H_{32}ClN_3OS$)

White solid. Yield: 85%. Mp: 214.2–215.6 °C. IR: = 3119, 2945, 2862, 1658, 1594, 1492, 1434, 678 cm^{-1}. MS(ESI (+)) *m/z*: 494 ($M+H^+$), 516 ($M+Na^+$). ^1H NMR (DMSO, 300 MHz): δ = 13.02(s, H, –CONH–), 7.55–8.09 (m, 4H, Ar–H), 6.81–7.19 (m, 3H, Ar–H), 2.33–2.81 (m, 2H, –CH–), 1.71–2.01 (m, 10H, –CH_2–), 1.42–1.52 (m, 12H, –CH_3). Calc. for $C_{28}H_{32}ClN_3OS$, %: C, 68.06; H, 6.53; N, 8.50. Found, %: C, 67.95; H, 6.72; N, 8.58.

2.3.5. N-[5-dehydroabietyl-[1,3,4]thiadiazol-2-yl]-p-nitryl benzamide (**2e** $C_{28}H_{32}N_4O_3S$)

Yellow solid. Yield: 81.4%. Mp: 243.3–244.8 °C. IR: = 3138, 2930, 2859, 1668,1604, 1526, 1494, 1436, 1343, 665 cm^{-1}. MS(ESI (+)) *m/z*: 505 ($M+H^+$), 527 ($M+Na^+$). ^1H NMR (DMSO, 300 MHz): δ = 13.41 (s, H, –CONH–), 8.25–8.35 (m, 4H, Ar–H), 6.81–7.19 (m, 3H, Ar–H), 2.33–2.79 (m, 2H, –CH–, 1.71–2.01 (m, 10H, –CH_2–), 1.26–1.51(m, 12H, –CH_3). Calc. for $C_{28}H_{32}N_4O_3S$, %: C, 66.64; H, 6.39; N, 11.10. Found, %: C, 66.58; H, 6.66; N, 11.21.

2.4. X-ray crystal structure determination

The crystal structure of **2b** was determined by X-ray single crystal diffraction. XRD data were collected on a Enraf–NoniusCAD-4 diffractometer equipped with Mo Ka (λ = 0.71073 Å) at 293 K. A single crystal suitable for determination was mounted inside a glass fiber capillary. The structure of the title compound was solved by direct methods and refined by full-matrix least squares on F^2. All the hydrogen atoms were added in their calculated positions and all the non-hydrogen atoms were refined with anisotropic temperature factors. SHELXS 97 were used to solve he structure and SHELTL were used to refine the structure [25–27]. There are two molecules and 0.25 water molecule in the empirical formula. The crystallographic details are summarized in Table 1. The selected bond lengths and angles are shown in Table 2.

2.5. Antibacterial activity

The antibacterial activity of chemicals was estimated by the minimum inhibition concentration (MIC). The MIC of compound was determined by the flat panel double dilution method. Test strain selection pathogenic bacteria are as follows: *Escherichia coli, Staphyloccocus aureus, Klebsiella pneumoniae, Pseudomonas aeruginosa, Escherichia aerogenes,* and *Staphyloccocus epidermidis*. The samples were prepared

Table 1 Experimental crystallographic data for compound **2b**.

Empirical formula	$C_{29}H_{35}N_3OS$
Formula weight	452.56
Temperature	293 K
Wavelength	0.71073 Å
Crystal system monoclinic space group	C2
Cell dimensions	a = 22.356(5) Å b = 11.868(2) Å c = 21.071(4) Å β = 103.60(3)°
Volume	V = 5433.8(19) $Å^3$
Z	4
Density (calculated)	1.163 Mg/cm^3
$F(000)$	2042
Crystal size	0.30 × 0.20 × 0.10 mm
Theta range for data collection	2.20–25.02
Index ranges	$0 \leqslant h \leqslant 26$, $-14 \leqslant k \leqslant 14$, $-25 \leqslant l \leqslant 25$
Reflections collected	10267
Independent reflections	9996(R_{int} = 0.060)
Data/restraints/parameters	9996/0/593
Goodness of fit on F	0.986
Final R indices ($I > 2\sigma(I)$)	R = 0.073, wR = 0.191
Largest diff. peak and hole	0.55 and −0.40 e Å

Table 2 Selected bond lengths (Å) and bond angles (°) for compound **2b**.

Bond	Dist.	Bond	Dist.	Bond	Dist.
S(1)–C(21)	1.727(5)	S(1)–C(20)	1.734(5)	O(1)–C(22)	1.192(6)
N(1)–C(20)	1.294(6)	N(1)–N(2)	1.390(6)	N(2)–C(21)	1.279(6)
N(3)–C(22)	1.374(7)	N(3)–C(21)	1.385(7)	C(11)–C(20)	1.525(7)
C(1)–C(2)	1.394(7)	C(3)–C(4)	1.386 (8)	C(24)–C(25)	1.363(10)
C(28)–C(29)	1.370(10)				
Angle	(°)	Angle	(°)	Angle	(°)
C(21)–S(1)–C(20)	86.4(3)	C(20)–N(1)–N(2)	111.6(4)	C(21)–N(2)–N(1)	113.2(4)
C(22)–N(3)–C(21)	126.7(5)	N(3)–C(22)–C(23)	113.8(5)	O(1)–C(22)–C(23)	126.2(6)
O(1)–C(22)–N(3)	120.0(5)	N(1)–C(20)–C(11)	120.8(4)	N(1)–C(20)–S(1)	114.4(4)
C(11)–C(20)–S(1)	124.5(3)	N(2)–C(21)–N(3)	120.3(5)	N(2)–C(21)–S(1)	114.4(4)
N(3)–C(21)–S(1)	125.2(4)				

Symmetry transformation: a: $x, y+1, z$; b: $-x, y, -z+1/2$; c: $-x, -y+1, -z$.

at a concentration of 2560 mg/ml, then they were autoclaved at 121 °C for 30 min. Duplicate twofold serial dilutions of each sample were added into MH AGAR medium for final concentration of 2560, 1280, 640, 320, 160, 80, 40 and 20 mg/ml. Some samples were prepared by decuple diluting method. The cultured bacterium was diluted by sterile water to obtain a bacterial suspension. The density of the organism suspension was adjusted to 108 CFU/ml by adding sterile deionized water. After inoculation, the plates were incubated at 37 °C for 24 h. The colonies were counted and the MIC values were obtained. The MIC is defined as the lowest concentration of antibiotic at which there is no visible growth of the organism. Standard compounds Bromogeramine and Ampicillin Sodium were measured alone.

3. Results and discussion

3.1. Chemistry

Because $POCl_3$ can be dissolved in water, the reaction must be dry in the general synthetic procedure of 5-dehydroabietyl-2-azyl-1,3,4-thiadiazole. The reaction process of *N*-[5-dehydroabietyl- [1,3,4]thiadiazol-2-yl]-aromatic amide in dilute acid hydrolysis reaction will not be complete. So acid binding agents, such as sodium bicarbonate, sodium carbonate, three ethylamine, pyridine, are needed to improve the reaction yield.

All the synthesized compounds were characterized by using spectroscopic techniques like IR, MS, ^{1}H NMR and elemental analysis. The IR spectra of compounds **2(a–e)** exhibited characteristic moderate absorption bands at 2932–2867, 1755–1749 and 1660–1603 cm^{-1} which attributed to the stretching vibrations of the $–CH_3$, $–CH_2$, C═O and C═N, respectively. The characteristic C═C stretching bands at 1600–1450 cm^{-1}associated with the aromatic rings were observed in all the target compounds. In addition, compound **5d** exhibited a characteristic strong absorption band at 1098 cm^{-1} attributed to the stretching vibrations of the Ar–Cl; compound **5e** exhibited characteristic strong absorption bands at 1526 cm^{-1}, 1343 cm^{-1} attributed to the symmetrical stretching vibrations and the asymmetric stretching vibration of the $–NO_2$. The ^{1}H NMR spectra of all compounds **2(a–e)** exhibited characteristic signals at 6.83–7.82 ppm assigned to the aromatic protons and at 12.63–13.41 ppm assigned to the acylamino protons.

3.2. Structure analysis

Full crystallographic details of **2b** have been deposited at the Cambridge Crystallographic Data Center and allocated the deposition number of CCDC 683542. White crystals of **2b** suitable for X-ray analysis were obtained by solvent evaporation under room temperature. Its single-crystal structure was determined by X-ray crystallography. Only one molecular structure is shown in Fig. 1, in order to clearly see the structure of compound **2b**. As shown in Fig. 1, the molecular structure of **2b** contains four crystallographically unique six-membered rings: ring A (C1–C6), ring B (C1, C6-C10), ring C (C10, C9, C11–C14) and ring D (C24–C29). Their torsion angles show ring B and C exhibit chair and half-chair configuration, respectively, they form trans ring junction with two methyl groups (C15, C16) in axis positions. However, the six atoms in the other two six-membered rings are coplanar. The sum of O1–C22–N3, O1–C22–C23 and N3–C22–C23 angles is 360°, which indicates sp2 hybridization state of the C22 atom. Due to the p–π conjugating effect, the C22–N3 bond (1.374 (7) Å) is shorter than the typical C–N bond (1.47 Å) and approximates to the typical C═N bond (1.35 Å). Based on the shorter bond lengths of N1–N2 1.390(6) Å, C11–C20 1.525(7) Å, C21–N3 1.385(7) Å and C20–S1 1.734(5) Å, C21–S1 1.727(5) Å than ordinary N–N, C–C, C–N and C–S single bond, and longer bond lengths of C20–N1 1.294(6) Å and C21–N2 1.279(6) Å than ordinary C–N double bond, we can conclude that conjugation occurs between the two C–N double bonds. The title compound is a chiral molecule with three chiral centers in each molecule, in which C9, C10 and C11 exhibit R-, S-, R- absolute stereo-configuration, respectively. From the view of crystal packing (Fig. 2), it is found that weak inter-molecular π···π stacking interactions exist in the structure.

3.3. Biological evaluation

Five newly synthesized compounds were screened for their antibacterial activitives against six kinds of bacteria, namely,

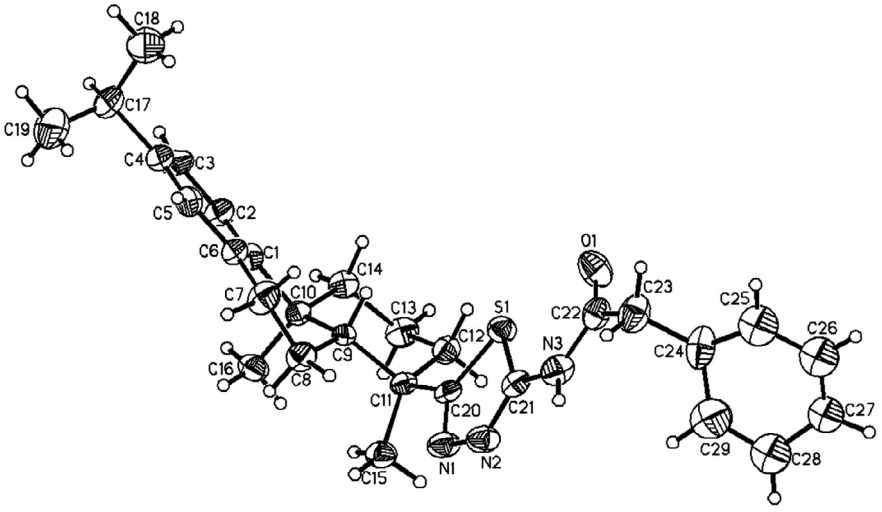

Figure 1 X-ray structure of compound **2b**.

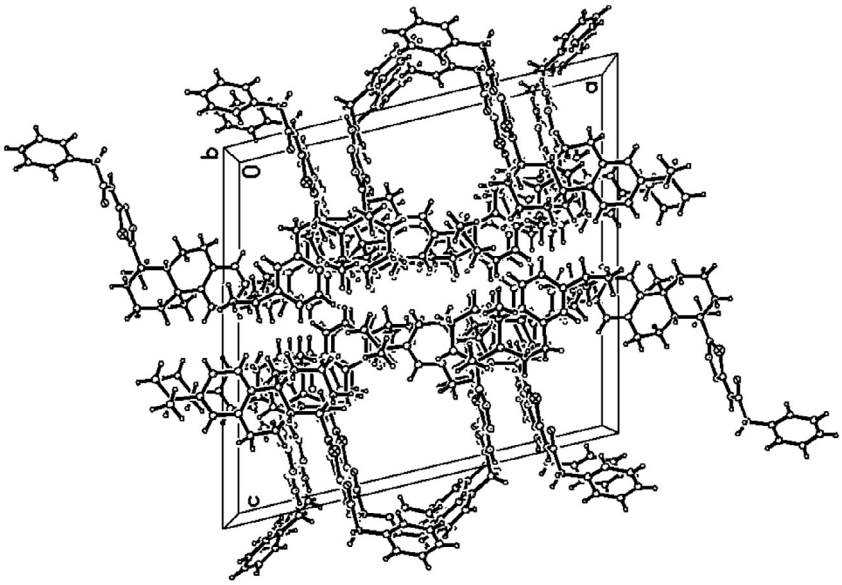

Figure 2 The packing diagram of compound **2b** the unit cell.

Table 3 Antimicrobial activities of the compounds **2(a–e)** (μg/mL).

Compound	A	B	C	D	E	F ①
2a	> 256	256	> 256	4	> 256	64
2b	> 256	> 256	> 256	8	64	> 256
2c	> 256	> 256	> 256	8	> 256	> 256
2d	> 256	> 256	> 256	8	> 256	> 256
2e	> 256	> 256	> 256	8	> 256	> 256
Bromogeramine	256	8	64	8	128	8
Ampicillin	64	4	64	> 256	> 256	8

A: *Escherichia coli* B: *Staphyloccocus aureu* C: *Klebsiella pneumoniae* D: *Pseudomonas aeruginosa* E: *Escherichia aerogenes* F: *Staphyloccocus epidermidis*.

E. coli (CMCC-44102), *S. aureus* (CMCC-26003), *K. pneumoniae* (GIM-1.279), *P. aeruginosa* (CMCC-10104), *E. aerogenes* (GIM-1.234) and *S. epidermidis* (CMCC-26069). Some of them were compared to standard Bromogeramine and Ampicillin Sodium. The MIC method [25] was used to assay the antibacterial activity. The results are summarized as MIC values in μg/mL in Table 3.

Nevertheless, compound **2a** showed efficacious antibacterial activity against Pseudomonas aeruginosa. The result implicated that blockage of the interaction of the aromatic ring group may be the main reason causing a decrease of the antibacterial activity because of the steric hindrance of aromatic ring para substituents. Therefore, changing the para substituents group of aromatic ring did not increase the antibacterial activity of these *N*-[5-dehydroabietyl-[1,3,4] thiadiazol-2-yl]-aromatic amide derivatives.

In conclusion, we have prepared a new series of *N*-[5-dehydroabietyl-[1,3,4]thiadiazol-2-yl]- aromatic amide derivatives. All compounds exhibit anti-bacterial activity against Pseudomonas aeruginosa, and the minimum inhibition concentration of these compounds is 4, 8, 8, 8, and 8 μg/ml respectively. Comparing with the minimum inhibition concentration of 8 μg/ml of the commercial compound bromogeramine and > 256 μg/ml of Ampicillin Sodium, the synthesized compounds obviously show anti-bacterial activity.

4. Conclusion

In summary, a series of *N*-[5-dehydroabietyl-[1,3,4]thiadiazol-2-yl]-aromatic amides derivatives were synthesized from dehydroabietic acid. Their structures were characterized by IR, $_1$H NMR spectroscopy and elemental analysis. All compounds exhibited highly effective activities against Pseudomonas aeruginosa. Compound **2b** showed efficacious antibacterial activities against *E. aerogenes*. Further investigation of this type of compound is also in progress now.

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