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Synthesis and Characterization of Novel Benzothiazole-Based Schiff Bases Derived from Substituted Benzaldehydes

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Abstract : *Schiff bases are an important class of organic compounds that have attracted considerable attention in synthetic and medicinal chemistry due to their versatile structural features and diverse applications. In the present study, a series of benzothiazole-based Schiff base derivatives (3a–3d) were synthesized through the condensation reaction of 2-hydrazinyl-1,3-benzothiazole with appropriately substituted benzaldehydes using a conventional reflux method. The reactions were carried out in ethanol in the presence of a few drops of acetic acid as a catalyst, and the progress of the reactions was monitored by thin-layer chromatography (TLC). The synthesized compounds were isolated, purified by recrystallization and characterized using elemental analysis and spectroscopic techniques, including FT-IR, ^1H NMR and mass spectrometry. The FT-IR spectra indicated the presence of characteristic functional groups, while the ^1H NMR spectra exhibited aromatic proton signals in the range of δ 6.82–8.48 ppm, a methyl proton signal at δ 2.49 ppm and a phenolic hydroxyl proton signal at δ 14.02 ppm. Furthermore, the mass spectral data were consistent with the proposed molecular structures. The successful synthesis and structural characterization of the target compounds demonstrate the effectiveness of the conventional condensation approach for the preparation of benzothiazole-based Schiff base derivatives.*

Keywords: Substituted benzaldehyde, 2-hydrazinyl-1,3-benzothiazole, spectral Analysis, Biological activity

I. INTRODUCTION

In recent years, Schiff bases are widely used scaffolds prepared via condensation product between primary amines and carbonyl compounds (aldehydes or ketones) which gives peculiar imine linkages [1-2]. Because of their straightforward preparation, structural flexibility, high stability and strong chelating capacity, imines have gained interest of researchers and found wide uses in organic, material sciences, metal complexes, corrosion inhibitors, sensors, optical properties, catalysts and pharmaceutical applications [3-7]. The presence of C=N imine functional group is accountable for it's their chemical and biological applications. Schiff base are fascinating molecules that have been found to have wide bioactivities. Antimicrobial, antitumor, anticonvulsant, antioxidant, anti-inflammatory, antipyretic are among them [8 10]. Aromatic aldehydes, mainly those containing phenolic -OH groups, are used are key reactant for Schiff base preparation. Salicylaldehyde and salicylaldehyde based compounds gives important mediator in different areas of industries as well as science, with wide range of biological applications [11-12]. The C=N (imine) along with OH group can act as a strong doner of electron pair towards metal centres and forms bidentate or multidentate coordination compounds and enhancing pharmacophore activity [13-14]. For synthesis of Schiff base ligand, selection of amine is also equally important which is responsible for the ligand stability, structural arrangement, and reactivity. Here we have selected 2-hydrazinyl-1,3-benzothiazole, which is fused heterocyclic compound with S, N atoms. By considering wide

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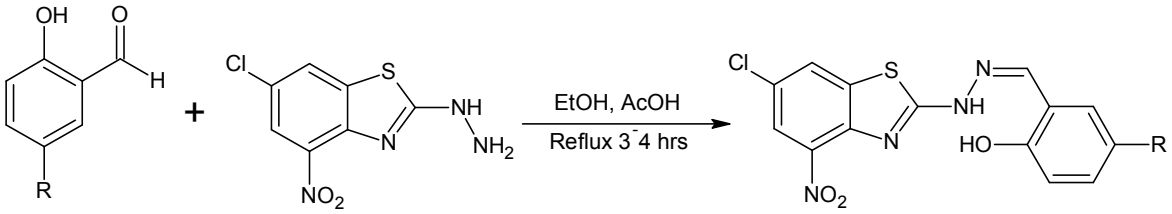
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range of biological activities of Schiff bases and metal complexes in the present research article we have synthesized some Schiff bases by conventional technique and all synthesized Schiff bases were evaluated for biological activity.

Materials and methods

All the chemical used in the synthesis are used were of A. R. grade. Melting points were determined in an open capillary tube and uncorrected. IR spectra were recorded in KBr on a Perkin-Elmer spectrometer. 1H NMR spectra were recorded on a Gemini 400 MHZ instrument in CDCl3 as solvent and TMS as an internal standard. The mass spectra were recorded on EI SHIMADZU-GC-MS spectrometer. Elemental analysis was carried out on Carlo-Erba mass analyzer. The purity of product was checked by thin layer chromatography (TLC) on Silica gel.

Reaction



R=H,Cl,Br,I,NO_2

Table 1. Physical data of Schiff Bases (3a-3d)

Entry	R	Colour	M.P. 0C	Yield(%)
3a	H	Yellow	121-122	64
3b	Cl	Pale Yellow	140-141	78
3c	Br	Yellow	171-172	62
3d	NO2	Yellow	180-182	76

General procedure for synthesis of Schiff bases

Equimolar quantities of substituted benzaldehyde and 2-hydrazinyl-1,3-benzothiazole amines were dissolved in ethanol (15 ml) acetic acid (0.3 ml) was added and refluxed for 3-4 hrs. After completion of reaction (monitored on TLC), the reaction mixture was cooled and poured in cold water, solid product was formed then separated out. Solid was filtered, washed with water and recrystallized from ethanol to give corresponding Schiff bases 3a-3d. M. P. yield and analytical data of Schiff bases are given in Table-1.

Results and Discussion

Schiff bases (3a-3d) were synthesized by condensation of substituted benzaldehyde with 2-hydrazinyl-1,3-benzothiazole. substituted benzaldehyde and 2-hydrazinyl-1,3-benzothiazole were dissolved in ethanol few drops of acetic acid added and refluxed for 3-4 hrs (monitored on TLC). Reaction mixture on cooling to room temperature on pouring in cold water solid separated. Solid was filtered, washed with cold water and crystallized with ethanol. The structure of the Schiff bases were assigned on the bases of elemental analysis and spectral data. IR spectra of Schiff bases showed characteristic band in the region 1629 cm-1 due to C=N stretching vibration and band around 1452 cm-1due to aromatic stretching. 1H NMR spectra of Schiff bases showed multiplet in the region of 6.82-8.48 due to aromatic proton. A singlet of methyl group was observed at 2.49 and phenolic hydroxyl group as a singlet at 14.02. Mass spectra of the Schiff bases were in agreement with its suggested structure.

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2-[(1E)-[2-(6-Chloro-4-nitro-1,3-benzothiazol-2-yl)hydrazine-1-ylidene]methyl] phenol (3a)
Yield-64%, Colour-Yellow, M.P.121-1220C. FT-IR (KBr,cm-1):3434(vOH), 3125(vNH),1629(vC=N azomethine), 1525(vC=N benzothiazole),1452(vC=C), 1018(vC-N-C), 670(vC-S-C).1H NMR(400 MHz, CDCl3): δ14.02(s,1H,Ar-OH), δ9.7(s,1H NH), δ8.48-6.82 (m,7H,Ar-H), . Analytical calc. for C14H9O3ClN4S(348.76): C 48.21, H 2.60, O 13.76, N 16.06, S 9.19, Cl 10.16 : found C 48.20, H 2.58, N 16.04.

4-Chloro-2-[(1E)-[2-(6-Chloro-4-nitro-1,3-benzothiazol-2-yl)hydrazine-1-ylidene]methyl] phenol (3b)
Yield-78%, Colour-Pale Yellow, M.P.140-1410C. FT-IR (KBr,cm-1):3434(vOH), 3125(vNH),1629(vC=N azomethine), 1525(vC=N benzothiazole),1452(vC=C), 1018(vC-N-C), 670(vC-S-C).1H NMR(400 MHz, CDCl3): δ14.02(s,1H,Ar-OH), δ9.7(s,1H NH), δ8.48-6.82 (m,6H,Ar-H),. Analytical calc. for C14H8O3Cl2N4S(383.20): C 43.88, H 2.10, O 12.53, N 14.62, S 8.37, Cl 18.50 : found C 43.86, H 2.08, N 14.60.

4-Bromo-2-[(1E)-[2-(6-Chloro-4-nitro-1,3-benzothiazol-2-yl)hydrazine-1-ylidene]methyl] phenol (3c)
Yield-62%, Colour-Yellow, M.P.171-1720C. FT-IR (KBr,cm-1):3434(vOH), 3125(vNH),1629(vC=N azomethine), 1525(vC=N benzothiazole),1452(vC=C), 1018(vC-N-C), 670(vC-S-C).1H NMR(400 MHz, CDCl3): δ14.02(s,1H,Ar-OH), δ9.7(s,1H NH), δ8.48-6.82 (m,6H,Ar-H), . Analytical calc. for C14H8O3BrClN4S(427.66): C 39.32, H 1.89, O 11.22, N 13.10, S 7.50, Cl 8.29, Br 18.68 : found C 39.30, H 1.87, N 13.08.

4-Nitro-2-[(1E)-[2-(6-Chloro-4-nitro-1,3-benzothiazol-2-yl)hydrazine-1-ylidene]methyl] phenol (3d)
Yield-76%, Colour-Yellow, M.P.180-1820C. FT-IR (KBr,cm-1):3434(vOH), 3125(vNH),1629(vC=N azomethine), 1525(vC=N benzothiazole),1452(vC=C), 1018(vC-N-C), 670(vC-S-C).1H NMR(400 MHz, CDCl3): δ14.02(s,1H,Ar-OH), δ9.7(s,1H NH), δ8.48-6.82 (m,6H,Ar-H). Analytical calc. for C14H8O5ClN5S(393.76): C 42.70, H 2.05, O 20.32, N 17.79, S 8.14, Cl 9.00 : found C 42.68, H 2.00, N 17.77.

Antimicrobial activity
All the newly synthesized compounds were screened in vitro antibacterial activity evaluated against 24hr culture of different bacterial strains such as Staphylococcusaureus, Streptococcus oyogenes (Gram +ve) and Escherichia coli, pseudomonasaeruginosa (Gram -ve) at a concentration 50 µg ml-1. The cultures were diluted with 5% of autoclaved saline and the final volume was adjusted to a concentration of approximately 105-106 CFU ml-1. The synthesized compounds were diluted with acetone for the antibacterial biological assay for agar disc diffusion method. The liquid form of test compounds was soaked on to a disc (5mm) and then allowed to air dry, such that the disc became completely saturated with the test compound. The saturated chemical discs were introduced into the upper layer of the medium evenly loaded with the bacteria and incubated at 370Cfor 24 to 48 hrs. all the experiments were performed in triplicate and the result are expressed as zone of inhibition of the synthesized compounds (3a-3d) was compared with zone inhibition of standard antibiotics Ofloxacin (50 µg ml-1). From the screening studies (Table 2), it is evident that the synthesized Schiff bases derivatives 3a,3b, 3c, 3d showed good antibacterial activity against all the tested organisms. It was further observed 3d was better over the all other compounds. This showed best activity near to that of standard drug. This observation leads to conclusion that halogen substituted Schiff bases showed higher activity against bacterial strain tested.

Table 2. Antibacterial activity of Compounds

Sr. No.	Compounds	Diameter of zone of inhibition (mm)			
		Gram (+ve) bacteria		Gram (-ve) bacteria	
		S.aureus	S.pygenes	E.coli	P.Aeruginosa
1	3a	18	17	14	17
2	3b	19	20	18	21
3	3c	20	21	21	20

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4	3d	27	28	27	26
5	Standard	30	29	28	28
6	DMSO	-	-	-	-

II. CONCLUSION

In the present work, we synthesized some schiff bases derivatives from different substituted benzaldehyde and 2-hydrazinyl-1,3-benzothiazole. The newly synthesized compounds were obtained in good yield and confirmed by spectral analysis. The antibacterial data revealed that electron donating and halogen containing Schiff bases showed higher activity against bacterial strain tested. It also revealed that all compounds showed good to moderate compared to standard drug.

Conflict of interest

There is no conflict of interest in the present study

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