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Research Article

MICROWAVE ASSISTED SYNTHESIS AND CHARACTERIZATION OF SCHIFF BASE OF 2-AMINO-6-NITROBENZOTHIAZOLE

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ABSTRACT

The main aim of this work was to synthesise a new Schiff base (2,4-diiodo-6-{[(6-nitro-1,3-benzothiazol-2-yl) imino] methyl} phenol)derivative. The above mentioned derivative was prepared by performing microwave-induced condensation reactions of 2- amino-6-nitro benzothiazole and 3,5-diiodosalicylaldehyde.The method used in the study is eco friendly and provides many benefits such as to straightforward work-up procedure, short reaction times, non-hazardous and good yield of products. The newly synthesized compound has been characterized by elemental analysis, IR, ^{1}H NMR, MS and MS-spectroscopy in the study.

Keywords: Microwave irradiation, Schiff base, Conventional, Benzothiazoles, 3, 5-diiodosalicyldehyde.

INTRODUCTION

The microwave induced enhancement of organic reactions is a branch of green chemistry. The application of microwave-assisted synthesis in organic, pharmaceuticals and coordination chemistry continues to develop at an astonishing pace. Microwave assisted organic synthesis (MAOS) has become a new and speedily emergent area in the synthetic organic chemistry. This eco-friendly technique is based on the observation that some chemical reactions proceed much faster and with higher yields under microwave irradiation as compared to conventional heating[1]. In microwave assisted organic synthesis, to escape accidents low boiling, toxic and poisonous solvents are frequently avoided. The use of microwave irradiation for the synthesis of drugs and organic compounds have proved to be effective, safe and environmentally nonthreatening techniques with shorter reaction time[2]. The prominent features of microwave irradiation technique are shorter reaction times, simple reaction conditions and enhancements in yields[3]. Compounds containing azomethine group (-HC=N-) are known as Schiff base[4]. They are condensation products of ketones or aldehydes with primary amines and were first reported by Hugo Schiff in 1864[5]. Schiff bases are biological active compounds they possess a lot of biological activities such as anticancer[6], plant growth inhibitors[7] insecticidal[8], antidepressant9, antibacterial[10], anti-inflammatory [11], anti-tuberculosis[12], antimicrobial[13] and anticonvulsant activity[14].

Schiff bases have number of applications viz., synthetic use, identification, detection and determination of aldehydes or ketones, purification of carbonyl or amino compounds, or protection of these groups during complex or sensitive reactions[15].

Benzothiazoles is heterocyclic compound its ring made from thiazole ring fused with benzene ring. Benzothiazoles are bicyclic ring system. In the 1950s, a number of 2-amino benzothiazoles were intensively studied as central muscle relaxants and found to interfere with glutamate neurotransmission in biochemical, electrophysiological and behavioural experiments [16]. Benzothiazoles ring found to be possessing antimicrobial activities such as antibacterial [17], antimicrobial[18], antidiabetic[19], antitumor[20], anti- inflammatory[21].

The aim of the present study was to prepare, the Schiff base derived from 2-amino -6-nitro benzothiazole and3,5 diiodosalicyldehyde under microwave irradiation. This Schiff base are identified by IR, ^{1}HNMR, GC-MS spectral and elemental analysis.

MATERIALS & METHODS

All the used chemicals and solvents were of Analytical grade. All the reagents used for the preparation of the Schiff bases were obtained from Sigma Aldrich. Melting point was determined in open capillary and is uncorrected. The IR studies of the schiff were recorded with 3000 Hyperion Microscope with Vertex 80 FTIR System in KBr pellets or Nicol phase from 4000 cm $^{-1}$ to 200 cm^{-1} at SAIF IIT Mumbai. Elemental analysis was carried out on Flash EA 1112 series Elemental Analyser System from IIT, Mumbai. The mass spectra of a Schiff base in this study were recorded at SAIF IIT Madras by (GC-MS Spectrometer Model Joel GC Mate. 1HNMR spectra in $CDCl_3$ were recorded on NMR spectrophotometer 500 MHz FT NMR Spectrometer at SAIF IIT Madras.

Conventional Method for Synthesis of Schiff Base

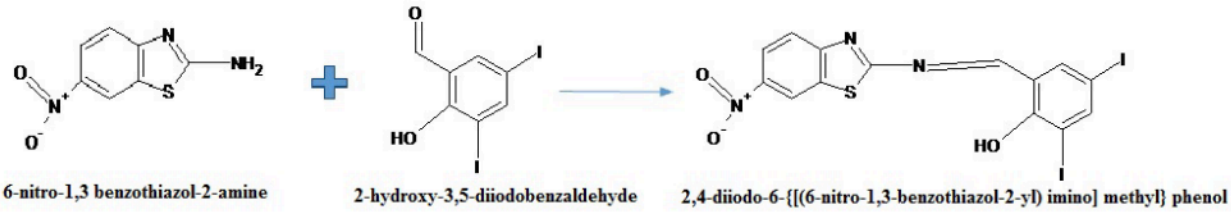
Ethanolic solution of 3,5-diiodosalicyldehyde (0.01 mol) were added drop wise to a ethanolic solution of 2- amino- 6- nitro benzothiazole (0.01 mol). The mixture was refluxed on water bath for 2 hrs. then cooled to room temperature and yellow product

was filtered. The product was recrystallized from methanol. Yield: 38%. The Schiff base ligand exists in crystalline or amorphous form, light yellowish in colour and are stable to air and moisture.

Microwave assisted Synthesis of Schiff base

The Synthesis of Schiff base is schematically presented in (scheme 1).

2-amino -6-nitro benzothiazole (0.01 mol) and 3,5-diiodosalicyldehyde (0.01 mol), and were mixed thoroughly in ethanol and small amount of glacial acetic acid was added and were taken in Erlen Meyer flask capped with a funnel placed in a microwave oven and irradiated an interval of 1 min at 450W for about 8-10 min. After completion the reaction, the reaction mixture was allowed to attain room temperature and solid separated was filtered. The resulting product was then recrystallized with ethanol, finally dried under reduced pressure over anhydrous $CaCl_2$ in a desiccator. Yield: 78%.



Scheme 1: Synthesis of Schiff baseNO_2

RESULTS AND DISCUSSION

As a result of the study, an efficient, solvent free and microwave assisted method is developed for the synthesis of Schiff base derivative (2,4-diiodo-6-{[(6-nitro-1,3-benzothiazol-2-yl) imino] methyl} phenol) which gives excellent yield with very shorter reaction time, as compared to conventional methods and yields have been also improved from 30-46to 76-80%.

Physical properties

The details of physical properties of the Schiff base are tabulated in (table 1).

Table 1: The comparative results of conventional and microwave methods, analytical and physical data of the compounds

S.No	Compound	Reaction time	Yield %	Melting point	Elemental ANALYSIS		
		CM MW	CM MW		C%	H%	N%
1	BENZ-S	2 (10)	38% 78%	158-160C^0	30.51	1.28	7.62

CM = Conventional method, time in hours; MW = Microwave method, time in minutes

IR Spectral Studies

Disappearances of carbonyl and amine group peaks from IR spectrum indicated formation of Schiff base. In the Schiff base strong peaks of carbonyl near 1723nm and amine near 3315nm were observed. Both of these peaks were absent in the IR spectra of Schiff base. In addition to that another peak was observed near 1654nm which is an indication of azomethine (CH=N). This reflects that amino acid and aldehydes which are the substrate for synthesis have been converted into Schiff base i.e. 2-amino 6-nitro benzothiazoles and,3,5 diiodosalicyldehyde. The data of the IR spectra of investigated Schiff base are listed in Table 2.

Table 2: Observed IR bands (cm-1) of Schiff base ligands

Compound	V,(O-H)	V,(C=N)	V,(C-S-C)	V.(N-O)	V,(C-O)
BENZ-s	3454	1654	732	1324	1275

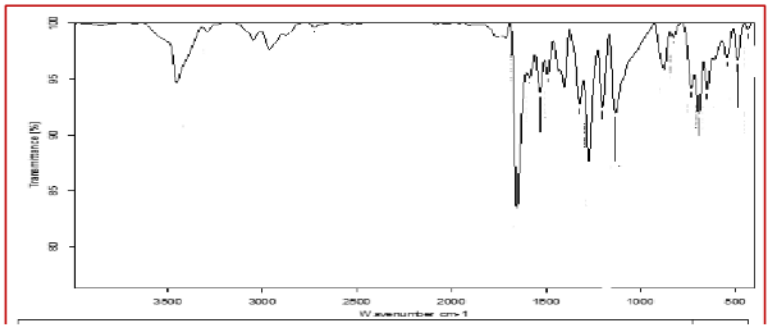


Figure 1:FTIR spetrum of schiff base

^{1}HNMR Spectral Studies

The 1H-NMR Spectra of schiffbase are given some signals which are summarised in Table 3.

Table 3: Observed ^{1}HNMR Peaks (ppm) of schiff base

S.No	Compound	**H for** from azomethine group	H from aromatic group	H from Phenolic proton
1	**BENZ-S**	**9.71 ppm**	**7.8-8.2ppm**	**11.74ppm**

The H-NMR spectrum for schiff base showed a peak at11.74 ppm (s, 1H, –OH), a peak at 9.71ppm (s, 1H, N=CH), The multi signals within the8.2-7.8 ppm range are assigned to the aromatic protons of both rings. The free NH2 protons usually show a broad singlet peak in a region at 4-6ppm. This NH_2 signal is absent in the observed spectra of Schiff bases which indicates the formation of the Schiff base[22].

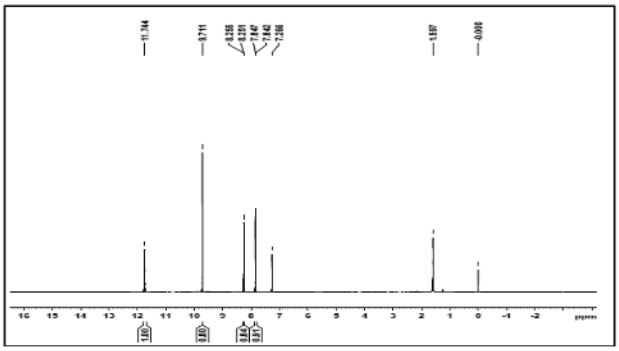


Figure 2: ^{1}HNMR spectrum of Schiff base

Mass Spectral studies

The mass spectrum of the schiff base showed the molecular ion peak at m/z 551 that corresponds to the molecular weight of the Schiff base.

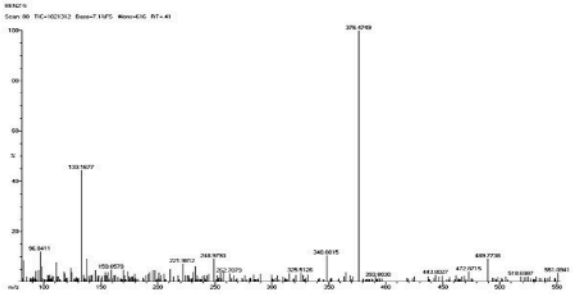


Figure 3:GC-MS spectrum of Schiff base

CONCLUSION

In this paper, we described new Schiff base which have been synthesized using condensation of 2-amino 6-nitro benzothiazoles and 3,5-diiodosalicyldehyde efficiently in an alcoholic medium using acetic acid with excellent yields under microwaves irradiation and characterized by various physicochemical and spectral analyses. In the result of microwave assisted synthesis of schiff base (2,4-diiodo-6-{[(6-nitro-1,3-benzothiazol-2-yl) imino] methyl} phenol), it has been observed that the reaction time decreased from hours to minutes and availability of the product within better yields compared to conventional method.

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