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Synthesis and Spectral Characterization of Schiff Base of 4-Amino- 3, 5-Dimethyl Isoxazole

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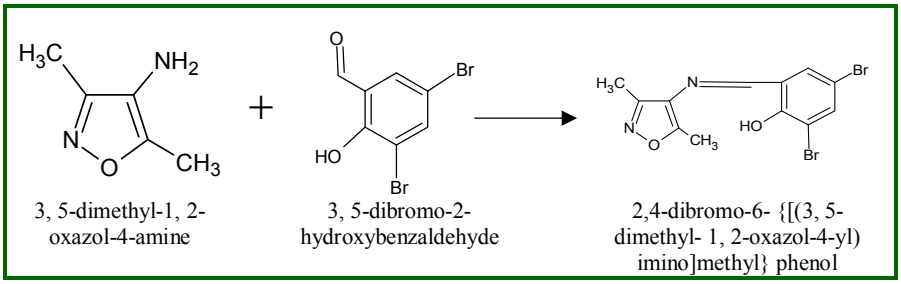
ABSTRACT

Microwave assisted synthesis are less energy consuming than conventional method and are rapidly becoming popular as green and sustainable alternative to conventional processes. The present paper deals with the microwave assisted synthesis of Schiff base (2, 4-dibromo-6-{[(3, 5-dimethyl-1, 2-oxazol-4-yl)imino]methyl} phenol) by reaction of 4-amino-3,5-dimethyl isoxazole with 3,5-dibromo-salicyldehyde in ethanol. The structural features of the synthesized compound were confirmed by their physical properties and infrared, electronic elemental analysis MS-mass and ^{1}HNMR spectroscopic techniques.

Graphical Abstract



3, 5-dimethyl-1, 2-oxazol-4-amine

3, 5-dibromo-2-hydroxybenzaldehyde

2,4-dibromo-6- {[(3, 5-dimethyl- 1, 2-oxazol-4-yl) imino]methyl} phenol

Schematic diagram of Synthesis of Schiff base of 4-amino 3, 5 dimethyl isoxazole

Keywords: Microwave irradiation, Conventional, Schiff base, Ligand.

INTRODUCTION

Schiff base and their derivatives have wide applications in supramolecular chemistry, biochemistry, analytical chemistry, catalysis, dye industry and biological activities [1]. Schiff bases are imino group containing organic compounds they were discovered by a German chemist, Nobel Prize winner, Hugo Schiff in 1864 [2]. Schiff bases are used, e.g., in optical and electrochemical sensors, furthermore as in varied natural process ways, to enable detection of enhance property and sensitivity [3-5]. Schiff base derivative of heterocyclic compounds have gained importance in drug and pharmaceutical fields

due to a broad spectrum of biological activities like anticancer [6], plant growth inhibitors [7] insecticidal [8], antidepressant [9], antibacterial [10], anti-inflammatory [11, 12], anticonvulsant drug activity [13].

The use of microwave heating in organic synthesis has risen intensely within the last years, receiving widespread acceptance and changing into an important tool [14]. Microwave-assisted synthesis provides safe protocol with the advantage of increased reaction rates, higher yields of products, application of commercially available inexpensive reagents, and has provided the momentum for several chemists to modify from typical heating technique to microwave assisted chemistry [15].

Isoxazole is one of the bioactive heterocyclic compounds containing a ring with three carbon atoms, one oxygen atom and one nitrogen atom that exhibit a wide range of biological activities [16]. Studies on Schiff bases derived from 3-amino-5-methyl isoxazole and substituted salicylaldehyde had been stated in advance with the aid of traditional method [17]. The aim of the present study was to prepare, the Schiff base derived from 4 amino 3, 5 dimethyl isoxazole and 3, 5dibromosalicyldehyde under microwave irradiation. These Schiff bases are identified by IR, 1HNMR, GC-MS spectral and elemental analysis.

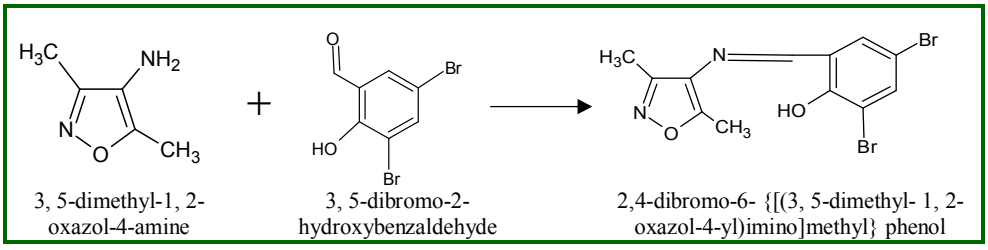
MATERIALS AND METHODS

Instrumentation: 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. ^{1}HNMR spectra in $CDCl_3$ were recorded on NMR spectrophotometer 500 MHz FT NMR Spectrometer at SAIF IIT Madras. The UV-Visible spectra were recorded on a Schimadzue UV spectrometer.

General procedure

Conventional Method for Synthesis of Schiff Base: Ethanolic solution of 3, 5 dibromoodosalicyl-dehyde (0.01 mol) was added drop wise to a ethanolic solution of 4 amino 3, 5-dimethyl isoxazole (0.01 mol). The mixture was refluxed on water bath for 2 h. The product was recrystallized from ethanol. Yield: 38-60%. 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: 4 amino 3, 5-dimethyl isoxazole (0.01 mol) and 3, 5-dibromosalicyldehyde (0.01 mol) were mixed thoroughly in ethanol and 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 5-8 min. After completion the reaction, the reaction mixture was allowed to attain room temperature and solid separated was filtered. Yield: 70-85%. The crude product was recrystallized from ethanol.



3, 5-dimethyl-1, 2-oxazol-4-amine + 3, 5-dibromo-2-hydroxybenzaldehyde → 2,4-dibromo-6- {[(3, 5-dimethyl- 1, 2-oxazol-4-yl)imino]methyl} phenol

Figure 1. Schematic diagram of Synthesis of Schiff base of 4-amino3,5 dimethyl isoxazole

RESULTS AND DISCUSSION

As a results of microwave-induced synthesis, it absolutely was ascertained that the observed synthesis of Schiff base (2, 4-dibromo-6-{[(3, 5-dimethyl-1,2-oxazol-4-yl) imino] methyl} phenol) was completed in an exceedingly short time with higher yields compared to the traditional method. Comparative study results obtained by microwave power-assisted synthesis; versus standard heating technique is that some reactions that needed 2-3 h, by standard technique was completed at intervals 8-10 min by the microwave irradiation technique, yields is improved from 35-58% to 72-85%.

Physical properties: The details of physical properties of the Schiff base are tabulated in table 1.

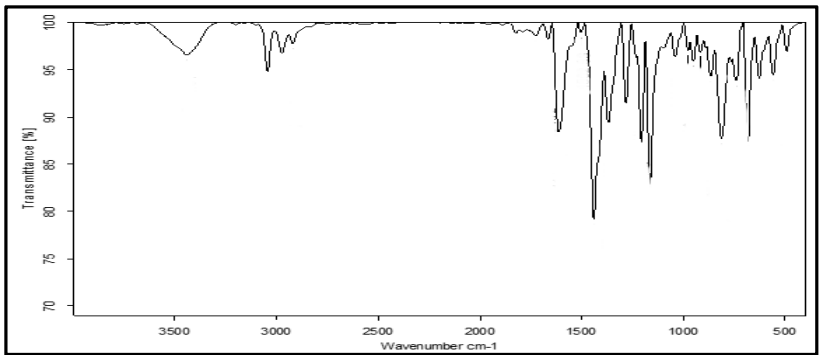
Table 1. The physical data of the Schiff base (2, 4-dibromo-6-{[(3,5-dimethyl-1,2-oxazol-4-yl)imino]methyl}phenol)

Compound	Physical appearance	Melting Point	Elemental analysis			m/z
			C	H	N	
ISO-S $C_{12}H_{10}Br_2N_2O2$	Light yellow color	200-205°C	(38.53) 38.08	(2.69) 2.503	(7.49) 7.191	374

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 1723 nm and amine near 3315 nm were observed. Both of these peaks were absent in the IR spectra of Schiff base [18]. In addition to that another peak was observed near 1613 nm 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. 4 amino3, 5-dimethyl isoxazole and 3, 5-dibromosalicyldehyde. The phenolic C-O stretch band is observed at 1276 cm^{-1} in the free ligand [19]. The data of the IR spectra of investigated Schiff base are listed in table 2.

Table 2. IR data of Schiff base and copper complex

Compound name	ν OH	ν C=N	ν C-O
ISO-S (Schiff base)	3441	1613	1281



Fiureg 2. IR spectra of Schiff base (2,4-dibromo-6-{[(3,5-dimethyl-1,2-oxazol-4-yl)imino]methyl} phenol).

^{1}HNMR Spectral Studies: The NMR spectra of Schiff base was recorded in CDCl3 solution, using tetramethylsilane (TMS) as internal standard. The H-NMR spectrum for Schiff base showed a peak at 13.68 ppm (s, 1H, –OH), a peak at 8.5ppm (s, 1H, N=CH). The multisignals within the 7.7-7.2 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 base which indicates the formation of the Schiff base [20]. The 1H-NMR Spectra of Schiff base are given some signals which are summarized in table 3.

Table 3. ^{1}HNMR Data of Schiff base (2,4-dibromo-6-{[(3,5-dimethyl-1,2-oxazol-4-yl)imino]methyl} phenol)

Compound code	-HC=N-	Aromatic Proton	Phenolic Proton	Two Methyl Protons
ISO-S (Schiff base)	8.5s	7.56-7.16.m	13.68.s	2.5-2.4,s,

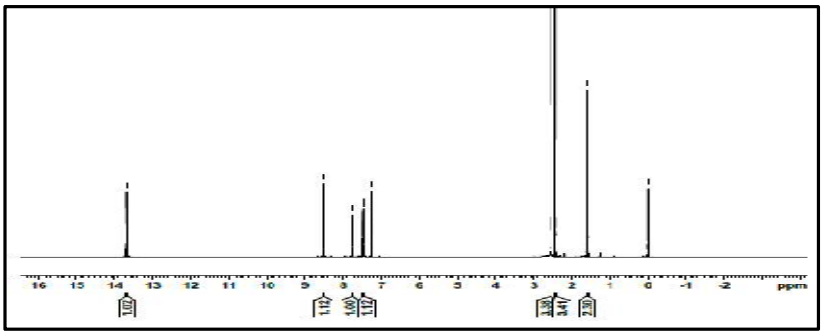


Figure 3. The ^{1}HNMR spectra of Schiff base (2,4-dibromo-6-{[(3,5-dimethyl-1,2-oxazol-4-yl)imino]methyl} phenol).

Electronic spectra of Schiff base: The electronic spectrum of the ligand has been measured in DMSO solution between 200-600 nm at room temperature. The electronic spectra of the ligand show series of bands within the range 200 to 250 nm and 300-350 nm and attributed to π–π* and n-π* transitions [2].

Mass Spectral studies: The mass spectrum of the Schiff base (2,4-dibromo-6-{[(3,5-dimethyl-1,2-oxazol-4-yl) imino]methyl} phenol)shows a molecular ion peak (m+.) at m/z 374 that corresponds to the molecular weight of the Schiff base.

APPLICATION

This method is useful for decreasing the reaction time from hours to minutes and availability of the product within better yields compared.

CONCLUSIONS

In this report, we described new Schiff base which have been synthesized using condensation of 4 amino 3, 5 dimethylisoxazole and 3, 5 dibromosalicyldehyde 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-dibromo-6-{[(3, 5-dimethyl-1,2-oxazol-4-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.

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Conflict of Interest: The authors declare that there is no conflict of interests regarding the publication of this paper Source of support: Nil

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