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Synthesis, Spectral Characterization and antifungal activity of a new Schiff base hydrazone derived from 2, 4-dinitrophenyl hydrazine

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ABSTRACT

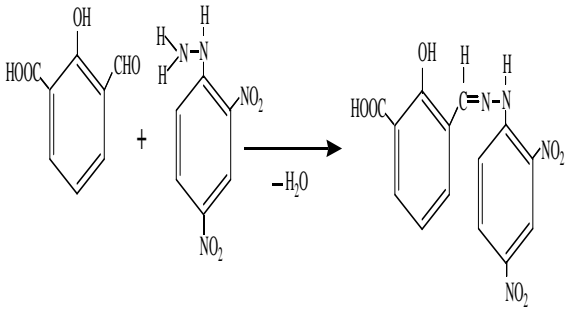
Hydrazone derivatives of carbonyl compounds constitute an important class of biologically active compounds. 3-aldehydosalicylic acid (3ASA) serves as precursor for the formation of hydrazone ligand. The condensation of 3-aldehydosalicylic acid with 2,4-dinitrophenyl hydrazine (2,4-DNPH) in the molar ratio 1:1, yield the corresponding hydrazone, ligand respectively. The structure of ligand was elucidated by elemental analyses, UV-VIS, FT-IR, ^{1}H, NMR and mass spectrometry. The Schiff base shows a significant antifungal activity against *Candida albicans, Aspergillus niger,* and *Penicillium sp*.

Keywords: Schiff Base; 2,4-dinitrophenyl hydrazine; 3-aldehydosalicylic acid; Antifungal; UV-vis; FT-IR; ^{1}H, NMR; and mass spectrometry.

1. INTRODUCTION

Schiff base play an important role in inorganic chemistry as they easily form stable complexes with most transition metal ions. The development in the field of bioinorganic chemistry has interest in Schiff base complexes, since it has been recognized that many of these complexes may serve as models for biologically important species [1-5]. The remarkable biological activity of acid hydrazides R-CO-NH-NH_2, a class of Schiff base, their corresponding aroylhydrazones, R-CO-NH-N=CH-R' , and the dependence of their mode of chelation with transition metal ions present in the living system have been of significant interest [6-12]. The coordination compounds of aroylhydrazones have been reported to act as enzyme inhibitors [13] and are useful due to their pharmacological applications [14-16]. Isonicotinic acid hydrazide (INH) is a drug of proven therapeutic importance and is used as bacterial ailments, e.g., tuberculosis [17]. Hydrazone derived from condensation of isonicotinic acid hydrazide with pyridine aldehydes have been found to show better antitubercular activity than INH [18,19]. Agarwal [18] investigated the coordinating ability of INH-derivatives with metal ions.

In the present investigation we describe the synthesis, characterization and antifungal activity of Schiff base hydrazone ligand. The structure of the newly hydrazone ligand was identified by elemental analyses, FT-IR, UV-VIS, mass and ^{1}H, NMR specta.



scheme. synthetic route for the preparation of ligand.

2. EXPERIMENTAL

2.1. Materials

3-aldehydosalicylic acid (3ASA) was synthesized according to duff and bills method [20]. Salicylic acid, Hexamethylenetetra ammine and 2,4-dinitrophenylhydrazine were obtained from Merck or BDH. All the chemicals used were of AR grade. Organic solvents (ethanol, absolute ethanol, methanol, diethylsther, acetone, dimethylformamide (DMF) and dimethylsulfoxide (DMSO) were reagent grade and were used without further purification.

2.2. Synthesis of the hydrazone ligand

The hydrazone ligand was prepared from a hot ethanolic solution of 3-aldehydosalicylic acid (3ASA) (1.66g, 10.00 mmol) in 20ml absolute ethanol and 2,4-dinitrophenylhydrazine (DNPH) (1.98g, 10.00 mmol) in 20 ml absolute ethanol,

respectively. The reaction was heated to reflux for 10 h, where orange precipitate was formed after cooling. The orange product was obtained by filtration and washed with absolute ethanol. Orange crystal was formed by recrystallization in ethanol.

2.3. Physical measurements

The elemental analysis (C, H, N) was carried on a truspec CHN/ CHNS analyzer. The FT-IR spectra (250-4000cm^{-1}) of the compound was recorded as KBr discs using Perkin Elmer-Spectrum RX-IFTIR instrument. The UV-vis spectra of the compound investigated was obtained on ECILUV5704SS UV-vis spectrophotometer in DMSO. The ^1H, NMR spectra of the ligand was obtained on WATERS, Q-TOF MICROMASS (LC-MS).

2.4. Antifungal activity

Pathogenic strains of *Asperigillus niger, Candida albicans* and *Penicillium sp* were obtained from Department of Microbiology ITM University, Gwalior. Schiff base was stored dry at room temperature and dissolved 20mg/ml in dimethylsulfoxide (DMSO). Antifungal activity of compound was evaluated by the agar disc-diffusion method. Sabarod's agar media (15 cm^3) kept at 45^oC was poured in the petri-dishes and allowed to solidify. Sterile, filter paper discs of 10mm diameter were impregnated with prepared Schiff bases (50µL) and were placed on to the media, seeded with fungus. The plates were then incubated at 27^oC for 1-7 days. At the end of period the inhibition zones formed on media were measured with a zone reader in millimeters

3. RESULTS AND DISCUSSION

The elemental anlysis of the ligand is in consistence with the molecular formula $C_{14}H_{10}N_4O_7$. Mass spectrum show the molecular ion peak at m/z 346, which corresponds to the molecular weight of the ligand. Hydrazone ligand soluble in solvents like DMSO and DMF but insoluble in some common organic solvents. The composition was established on the basis of elemental analysis, spectral analysis, melting point and colour (Table - 1) and are discussed in detail in the following section.

3.1. Infrared spectral study

The FT-IR spectrum of the ligand (Table-2) shows a strong bond at 3463cm^{-1} assigned to υOH of the phenolic group. The stretching vibration of NH group appears as weak band at 3300 cm^{-1}. The spectrum show also vibrational bands at 1585, 1609 and 1147 and 1130 cm^{-1} assigned for υ_s and υ_{as} of the C=N and N-N groups, respectively. The stretching vibration of $-NO_2$ group appear at 1330, -OH and C=0 streching of carboxylic group appears at 3087 and 1645cm^{-1}.

3.2. Electronic spectral studies

Electronic spectra of the compound in DMSO was scanned in the region 200-800 nm region. Electronic spectra of hydrazone Schiff base two prominent absorptions at 299 and 414 nm which can be assigned to π- π^* and n- π^* transition or which are attributed to change transfer transitions.

3.3. ^1H, NMR spectral studies

The ^1H, NMR spectrum of the ligand in $DMSOd_6$ showed two singals at δ= 9.8 ppm for the proton of the carboxylic COOH group and δ= 8.9 ppm for the NH group respectively. Two signals are also observed at δ= 8.2 ppm for the H-C=N group and δ= 7.8 ppm for the Phenolic OH group.

3.4. Mass spectral studies

The mass spectrum of the hydrazone, H_L, ligand revealed the molecular ion peak at m/e 346 which coincident with the formula weight and support the identity of the molecule.

3.5. Antifungal test

The antifungal study was carried out using the agar disc diffusion method. Three type of fungi viz. *Candida albicans, Aspergillus niger* and *Penicillium sp*. Were used as the test organisms. Based on the result, the ligand moderate activity towards the fungi, this might due to the NH group inside the ligand planging an important role in antifungal activity. This group is believed to impart the transformation reaction in biological activity of the ligand is due to either killing the microbes or inhibiting their multiplication by blocking their active site.

Table - 1: Elemental analysis, Colour, and Melting point of hydrazone ligand

Compound	Empirical formula	(M. wt.)	Elemental analysis (%)			Colour	Melting point (˚C)
			C	H	N		
H_L	$C_{14}H_{10}N_4O_7$	346	49.35 (48.55)	3.05 (2.89)	15.95 (16.18)	Orange	245 ˚C

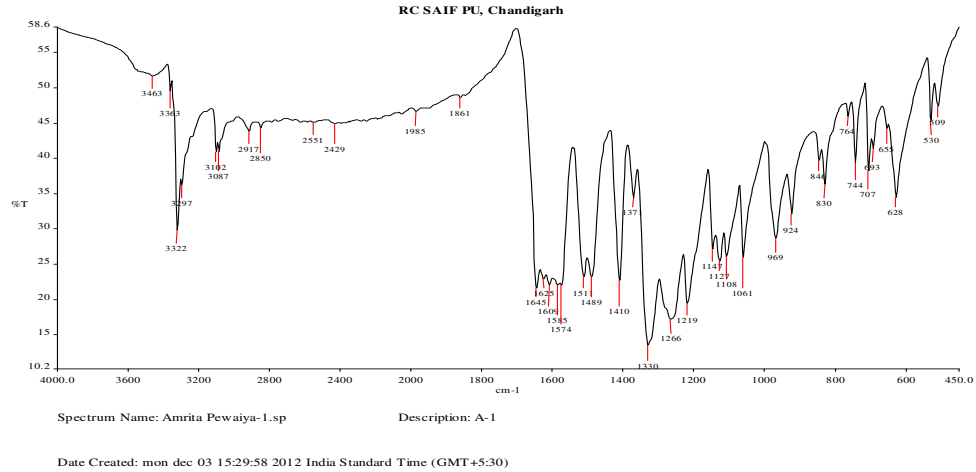


Figure - 2: FT-IR Spectra of Schiff base

Table - 2: Vibrational frequencies of Schiff base

Bond position	Assignment
υ(O-H)	3463
υ(N-H)	3300
$\upsilon(C=N)_s$ & $\upsilon(C=N)_{as}$	1585 & 1609
$\upsilon(N-N)_s$ & $\upsilon(N-N)_{as}$	1147 & 1130
$\upsilon(-NO_2)$	1330
υ(-OH)	3087
υ(C=O)	1645

4. CONCLUSIONS

The reaction of 3-aldehydosalicylic acid with 2, 4-dinitrophenyl hydrazine (2,4-DNPH) in the molar ratio 1:1, forming hydrazone, H_L ligand. The spectral (FT-IR, UV-vis, ^{1}H, NMR and mass) measurement of the ligand was used to determine structure. In vitro antifungal activity of synthesized compound show good result with an enhancement of activity with ligand.

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