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RESEARCH ARTICLE

SYNTHESIS, CHARACTERIZATION AND ANTIFUNGAL ACTIVITY OF HYDRAZONE SCHIFF BASE

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ABSTRACT

Hydrazones derived from aromatic acid hydrazides and aromatic or heterocyclic aldehydes have a wide variety of applications in many fields. 3-aldehydosalicylic acid (3ASA) serves as precursor for the formation of hydrazone ligand. The condensation of 3-aldehydosalicylic acid with Phenyl hydrazine hydrochloride in the molar ratio 1:1 yield the corresponding hydrazone, ligand respectively. The structure of ligand was elucidated by FT-IR, ^{1}H, NMR and mass spectrometry. The results are in consistent with bidentate chelation of ligand with azomethine nitrogen and ring nitrogen donors. The Schiff base show a significant antifungal activity against *Candida albicans*, *Aspergillus niger*, and *Penicillium sp*.

INTRODUCTION

A Schiff base, named after Hugo Schiff, is a compound with a functional group that contains a carbon-nitrogen double bond with the nitrogen atom connected to an aryl or alkyl group, not hydrogen (The" Gold Book" 1997). Schiff bases play an important role in inorganic chemistry. The importance of hydrazones and their derivatives arises from their wide use in many scientific fields. This use is due to presence of azomethine group (–NH–N=CH–) in their molecule. Hydrazones constitute an important class of biologically active drug molecules (Seleem *et al.,* 2011). which has attracted attention of medicinal chemists due to their wide range of pharmacological properties. Schiff base hydrazones are also interesting from the point of view of pharmacology. Hydrazone derivatives are found to possess antimicrobial (Vicini *et al.,* 2002), antitubercular (Kocyigit-Kaymakcioglu *et al.,* 2002), anticonvulsant (Ragavendran *et al.,* 2007) and anti-inflammatory (Rollas *et al.,* 2002) activities. Reaction of carbonyl with hydrazine gives a hydrazone. Hydrazine is more nucleophilic than a regular amine due to a presence of the adjacent nitrogen. Note the similarly to the formation of an oxime reaction. Hydrazones generally form a mixture of geometric isomers.

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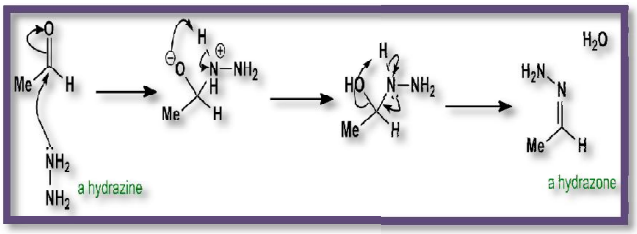
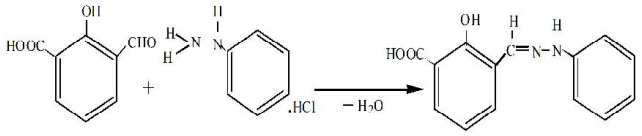


Scheme: Synthetic route for the preparation of Hydrazone

An interesting application of Schiff bases is their use as an effective corrosion inhibitor, which is based on their ability to spontaneously form a monolayer on the surface to be protected. Many commercial inhibitors include aldehydes or amines, but presumably due to the C=N bond the Schiff bases function more efficiently in many cases (Li *et al.,* 1999). The principal interaction between the inhibitor and the metal surface is chemisorption (Ashassi-Sorkhabi *et al.,* 2006). The inhibitor molecule should have centers capable of forming bonds with the metal surface by electron transfer. In such cases the metal acts as an electrophile and the inhibitor acts as a Lewis base. Nucleophilic centers, such as oxygen and nitrogen atoms, of the protective compound have free electron pairs which are readily available for sharing. Together with the atoms of the benzene rings they create multiple absorption sites for the inhibitor thus enabling stable monolayer formation (Quan *et al.,* 2001). This paper presents a new Schiff base with a potential biological activity resulted from the condensation of acid with hydrazine. These compound could also act as

valuable ligand. The structure of the Schiff base synthesized from 3-aldehydosalicylic acid with phenyl hydrazine hydrochloride is shown in scheme. The structure of the newly hydrazone ligand was identified by elemental analyses, FT-IR, mass and ^{1}H, NMR spectra



scheme. synthetic route for the preparation of ligand.

Experimental

Materials

3-aldehydosalicylic acid (3ASA) was synthesized according to Duff and Bills method (Duff *et al.,* 1932). Salicylic acid, Hexa methylenetetra ammine and Phenylhydrazine hydrochloride were obtained from Merck or BDH. All the chemicals used were of AR grade. Organic solvents (ethanol, absolute ethanol, methanol, diethylether, acetone, dimethylformamide (DMF) and dimethylsulfoxide (DMSO) were reagent grade and were used without further purification.

Synthesis of the hydrazone ligand

The hydrazone ligand was obtained by refluxing a mixture of 3-aldehydosalicylic acid (3ASA) and phenylhydrazine hydrochloride (PHH), (1:1 molar ratio) on a water bath for 8h. Ethanol (30 ml) was used as the solvent. After cooling, a light green precipitate formed, which was filtered, washed with ethanol and dried under vacuum over $CaCl_2$. (Melting Point 180^0C)

Physical measurments

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 disc using Perkin Elmer-Spectrum RX-IFTIR instrument. The ^{1}H,NMR spectra of the ligand was obtained on FT-NMR Spectrometer model Avance-II (Bruker). The mass spectra of the ligand was obtained on WATERS, Q-TOF MICROMASS (LC-MS).

Antifungal Testing

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^{o}C 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^{o}C for 1-7 days. At the end of period the inhibition zones formed on media were measured with a zone reader in millimeters.

RESULTS AND DISCUSSION

The elemental analysis of the ligand is in consistence with the molecular formula $C_{14}H_{12}N_2O_3$. Mass spectrum show the molecular ion peak at m/z 256, which corresponds to the molecular weight of the ligand. The composition was established on the basis of elemental analysis, melting point and colour (Table 1) and are discussed in detail in the following section.

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

Compound	Empirical formula	(M. wt.)	Elemental analysis, found Calculated (%) C	H	N	Colour	Melting point (^{0}c)
H_L	$C_{14}H_{12}N_2O_3$	256	66.35 (65.62)	3.98 (4.68)	10.12 (10.93)	light green	180^0

Infrared spectral study

The FT-IR spectrum of the ligand (Table 2) shows a strong bond at 3313cm^{-1} assigned to ^{v}OH of the phenolic group. The stretching vibration of NH group appears as weak band at 3212 cm^{-1}. The spectrum show also vibrational bands at 1608, 1488 and 1185 and 1157 cm^{-1} assigned for υ_s and $\upsilon_{as}$of the C=N and N-N groups, respectively. The stretching vibration of -OH and C=0 stretching of carboxylic group appears at 3004 and 1663cm^{-1}.

Table 2. Vibrational frequencies of Schiff base

S.No.	Bond position	Assignment
1.	υ(O-H)	3313
2.	υ(N-H)	3212
3.	υ(C=N) & υ(C=N)as	1608 & 1488
4.	υ(N-N) & υ(N-N) as	1185 & 1157
5.	υ(-OH)	3004
6.	υ(C=O)	1663

^{1}H, NMR spectral study

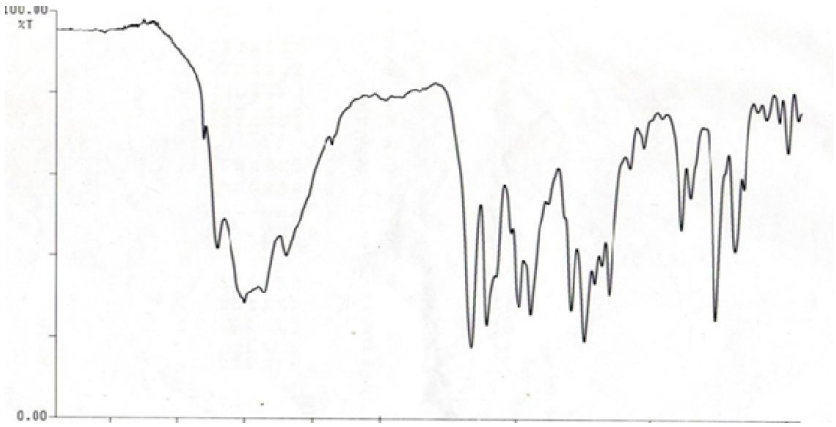
The ^{1}H, NMR spectrum of the ligand in DMSOd_6 showed two singals at δ= 10.3 ppm for the proton of the carboxylic COOH group and δ= 8.2 ppm for the NH group respectively. Two signals are also observed at δ= 7.8 ppm for the H-C=N group and δ= 6.9 ppm for the phenolic OH group.

Mass spectral study

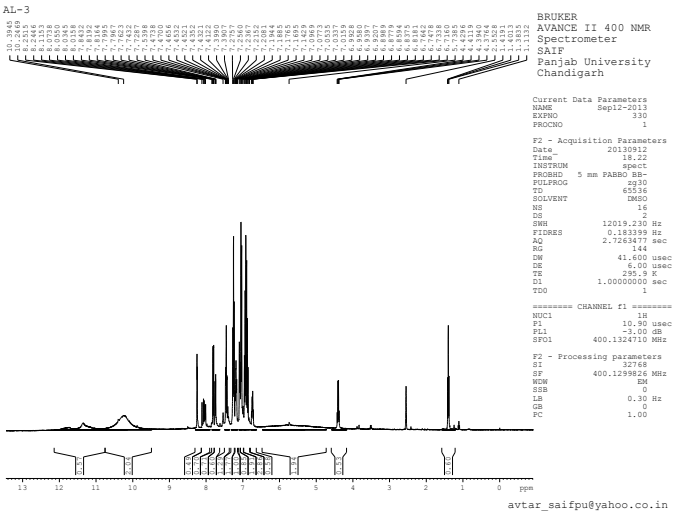
The mass spectrum of the hydrazone, H_L, ligand revealed the molecular ion peak at m/e 256 which coincident with the formula weight and support the identity of the molecule.

Antifungal activity

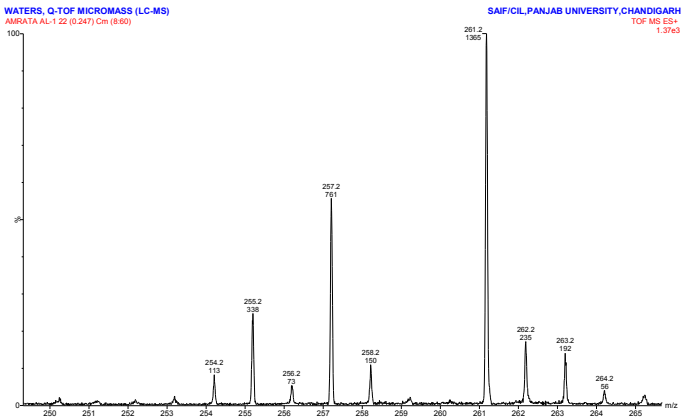
From the result obtained by the antifungal activity it is found that the Schiff base is more active against all tested fungi. Compound is the most potent candidate against all type of tested fungi. The greater activity of this compound is probably due to the presence of azomethine group.



FT-IR Spectra of hydrazone Schiff base



1H, NMR Spectra of hydrazone Schiff base



Mass spectra of hydrazone Schiff base

Conclusion

The work described in this paper involved the synthesis and spectroscopic characterization of hydrazone Schiff base. The synthesized compound appeared to have moderate antifungal activity against the three pathogens *Candida albicans*, *Aspergillus niger*, and *Penicillium sp*.

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