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Synthesis, characterisation and antibacterial activity of Schiff base, N-((5-methylthiophen-2-yl)methylene)-1-(pyridin-2-yl)methanamine

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Abstract:

A new novel Schiff base namely, N-((5-methylthiophen-2-yl)methylene)-1-(pyridin-2-yl)methanamine was synthesized by condensation of 2-picolyl amine with 5-methyl thiophene-2-carboxaldehyde. Schiff base was characterised by spectroscopic methods such as FT-IR, ^{1}H NMR, and ^{13}C NMR. The coordination behaviour of synthesised Schiff base with transition metal ions i.e., Cu(II) and Zn(II) was investigated and the Schiff base showed good coordination bond with metal ions. Antibacterial activity of the synthesised ligand and its metal complexes was evaluated against gram positive bacterial species such as Staphylococcus aureus, and Bacillus subtilis as well as gram negative bacteria such as Escherichia coli, and Pseudomonas aeruginosa. The Schiff base metal complexes showed higher antibacterial activity when compared to bare Schiff base.

Keywords: Schiff base, 2-picolyl amine, 5-methyl-thiophene-2-carboxaldehyde, metal complexes

1. Introduction

The chemistry of Schiff base ligands containing oxygen, nitrogen, and sulphur as donor atoms and Schiff base metal complexes is continued to be an interesting area of research because of their applications in the field of biological, industrial and material sciences. Under various reaction conditions, these ligands are known to coordinate to metal ions in various ways. The Schiff base ligands are derived by combining aldehydes and primary amines in a condensation process [1-5].

The biological activities of Schiff bases and their metal complexes are one of the primary fields of study, with the main goal of discovering safe and efficient therapeutic agents for the treatment of bacterial infections and malignancies. A wide range of biological and pharmacological activity can be found in Schiff base metal complexes. Transition metal complexes of Schiff base ligands bearing "O", "N", and "S" donor atoms are biologically important because of their known antibacterial, antifungal, anti-inflammatory [6-8], analgesic [7-12], anticonvulsant [13-16], antitubercular [17], antioxidant [18], and anthelmintic [19] properties. Schiff base transition metal complexes have also been utilised as biological models to better understand biomolecule structure and biological processes.

Numerous variants of the condensation products of imines with aldehydes or ketones, such as RCH=NR′, where R & R′ are alkyl and/or aryl substituents have been popularised since the Italian scientist, Hugo Schiff utilised imines to produce numerous Schiff bases. These Schiff bases have a variety of uses in organic research, including the building of new heterocyclic systems, the identification, detection, and determination of aldehydes and ketones, the purification of carbonyl or amino compounds, and the protection of these groups during the formation of complexes or other sensitive reactions [20]. They have other uses in various other fields, such as analytical chemistry [21], coordination chemistry [22–23], polymer industries [24] pigments and dyes [25] as well as vitamins and enzymes [26] for model biomolecules. These compounds are also used as insecticides and fungicides in agriculture.

In this study, we synthesized a new Schiff base, N-((5-methylthiophen-2-yl)methylene)-1-(pyridin-2-yl)methanamine derived from 2-picolyl amine and 5-methyl thiophene-2-carboxaldehyde. Further, Cu(II) and Zn(II) complexes of N-((5-methylthiophen-2-yl)methylene)-1-(pyridin-2-yl)methanamine were synthesized. The Schiff base and metal complexes were characterised using FT-IR, ^{1}H and ^{13}C NMR Spectroscopic methods. The biological activities of synthesised Schiff base and its metal complexes were evaluated for anti-bacterial activity towards gram positive and negative bacteria.

2. Materials and Methods

2.1 Synthesis of ligand

N-((5-methylthiophen-2-yl)methylene)-1-(pyridin-2-yl)methanamine was synthesized with modification of the procedure reported in the literature[21]. Briefly, 0.01 mole of 2-picolyl amine was dissolved in 15ml of ethanol and added 0.01 mole of 5-methyl thiophene 2-carboxaldehyde under constant stirring. The reaction mixture was refluxed for 5 hours at 65 ^{o}C to produce the Schiff base. The resulting Schiff base was cooled to room temperature, filtered, and then recrystallized in ethanol. The recrystallized Schiff base was dried at room temperature. The schematic representation for the preparation of Schiff base was given in Figure 1.

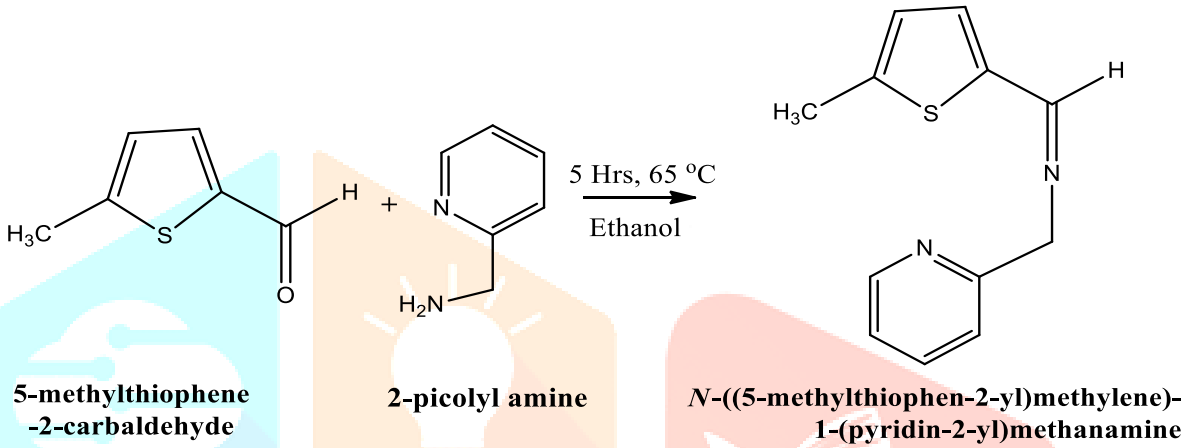

Figure 1. Schematic representation of Schiff base ligand

2.2 Synthesis of metal complexes

The metal salts and ligand were taken in 1:2 molar ratios in ethanol. A solution of the appropriate metal salt (0.001 mmol) (MX_2, Where M = Cu(II) (or) Zn(II); X = Acetates) dissolved in 15 ml ethanol was added to a hot ethanolic solution of the ligand (0.002 mmol). The mixture was refluxed for 6 hours at 60 ^{o}C. The complexes were precipitated by cooling the mixture to room temperature. The mixture was then filtered and washed with ethanol. Finally, the solid was dried in vacuum desiccators.

3. Characterization

3.1 FT-IR Spectral Studies

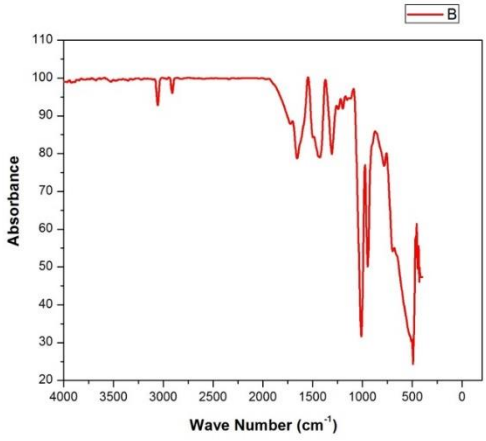

Figure 2. FT-IR Spectra of Schiff base

The FT-IR spectral image of the synthesized Schiff base was given in Figure 2. The absorption bands in the spectra at 2890 cm^{-1} and 3060 cm^{-1} can be assigned to the aliphatic and aromatic –C-H stretching vibrations. The imine group –C=N stretching vibration was seen at 1640 cm^{-1}. The aromatic –C=C stretching was seen at 1475 cm^{-1}. The –C-S stretching can be observed as

broad band around 500 cm^{-1} – 700 cm^{-1} range. The main vibrational frequencies in the IR range confirm the successful preparation of Schiff base.

3.2 ^{1}H NMR Studies

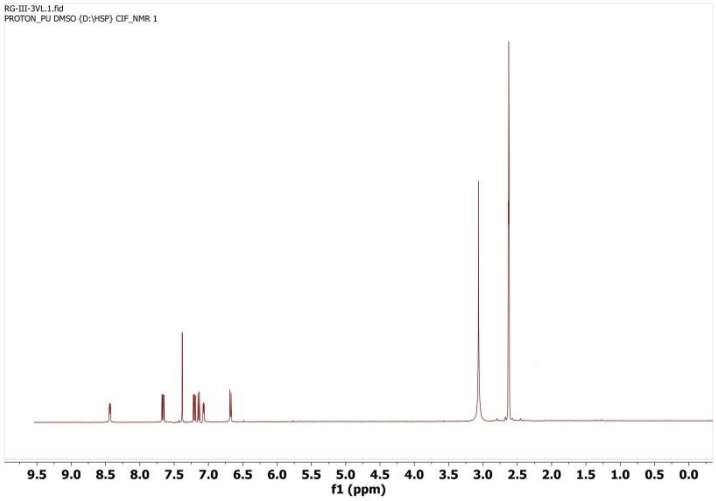

Figure 3. ^{1}H NMR Spectra of Schiff base

^{1}H NMR spectra of synthesized Schiff base, N-((5-methylthiophen-2-yl)methylene)-1-(pyridin-2-yl)methanamine was recorded and shown in Figure 3. The methyl hydrogen's of the thiophene ring were seen at 2.6 ppm with singlet peak and the methylene group hydrogen's that adjacent to the imine group was seen at 3.1 ppm with singlet peak. The pyridine and thiophene ring hydrogen atoms were seen in the range of 6.7 ppm to 8.5 ppm. The single hydrogen of the imine group was seen at 7.3 ppm.

3.3 ^{13}C NMR Studies

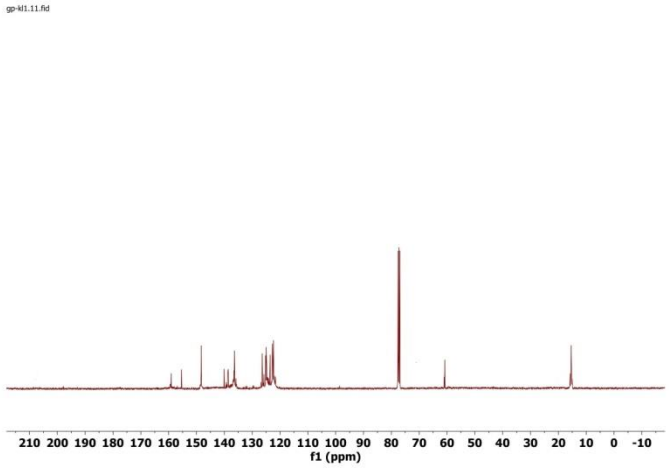

Figure 4. ^{13}C NMR Spectra of Schiff base

Figure 4 shows the ^{13}C NMR of the prepared Schiff base. The resonance peak around 15 ppm corresponds to the methyl carbon of the thiophene ring. The peaks at 124 ppm, 126 ppm, 142 ppm, and 144 ppm can be assigned to the four carbon atoms of the thiophene ring. The methylene carbon atom that adjacent to the imine group can be confirmed by the peak at 154 ppm. The carbon atoms of the pyridine ring can be the resonance peaks of 121 ppm, 137 ppm, 120 ppm, 148 ppm, and 158 ppm. Finally, the carbon atom of the imine group can is observed around 155 ppm.

4. Biological Applications

4.1 Antibacterial activity

The antibacterial activity of the prepared Schiff base, N-((5-methylthiophen-2-yl)methylene)-1-(pyridin-2-yl)methanamine and its metal complexes was evaluated against gram positive (*Staphylococcus aureus*, *Bacillus subtilis*) and gram negative (*Escherichia coli*, *Pseudomonas aeruginosa*) bacteria. Streptomycin was taken as the reference antibacterial. The solutions of the compounds and reference drug were dissolved in DMSO. From the Table 1, it is clear that the Schiff base complexes of Cu(II) and Zn(II) metals are more biologically active than the bare Schiff base ligand. Zinc complex of the Schiff base ligand showed higher activity than the other complex.

Table 1. Antibacterial activity of Schiff base and its metals complexes

Compound	*S. aureus*	*Bacillus*	*Pseudomonas*	*E. coli*
Ligand	11 mm	12 mm	10 mm	11 mm
$Cu(L)_2$	17 mm	15 mm	14 mm	18 mm
$Zn(L)_2$	19 mm	18 mm	17 mm	21 mm
Streptomycin	21 mm	26 mm	24 mm	24 mm

5. Conclusion

A new Schiff base was prepared by condensation of 2- picolyl amine and 5-methyl thiophene-2-carbaxaldehyde. The spectroscopic methods such as FT-IR spectroscopy, ^{1}H & ^{13}C NMR spectroscopy were used to elucidate structure and coordination with metal ions of the Schiff base. The biological activity of the prepared Schiff base and its metal complexes against gram positive (Staphylococcus aureus, Bacillus subtilis) and gram negative (Escherichia coli, Pseudomonas aeruginosa) microorganisms was tested. The Schiff base metal complexes were found to have higher biological activity than the bare Schiff base ligand.

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