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SYNTHESIS AND EVALUATION OF SCHIFF BASE LIGANDS AND THEIR METAL COMPLEXES OF CU(II) AND FE(III)

***[1]Mrs. Yogita M. Vispute, [2]Ms. Pallavi Shelar, [3]Ms. Nidhi Sawant, [4]Ms. Sonali Yadav, [5]Mr. Om Thakur, [6]Mr. Dilip Choudhary**

[1]Assistant Professor, Ideal College of Pharmacy and Research, Kalyan.

[2,3,4,5,6]Final Year Student, Ideal College of Pharmacy and Research, Kalyan.

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***Corresponding Author**
Mrs. Yogita M. Vispute
Assistant Professor, Ideal College of Pharmacy and Research, Kalyan.

ABSTRACT

A novel Schiff base ligand, N-(1-(2,4-dinitrophenyl) ethylidene) hydrazine, was synthesized and complexed with Cu(II) and Fe(III) ions. The ligand and its metal complexes were characterized using various analytical techniques, including UV-Visible spectroscopy, FTIR, and ^{1}H-NMR. The complexes showed high solubility in polar organic solvents. The antibacterial activity of the compounds was evaluated against Escherichia coli using the disc diffusion method. The Cu(II) complex exhibited the highest zone of inhibition, indicating strong antibacterial efficacy, followed by the Fe(III) complex and the free ligand. The results suggest that metal complexation enhances the antibacterial potential of Schiff base ligands. This study highlights the potential of metal complexes as antibacterial agents, with implications for the development of new therapeutic agents. The findings demonstrate the importance of metal complexation in enhancing biological activity.

KEYWORD: Mixed Lignd Metal Complex, Phenyl Hydrazine Schiff Base, 2, 4-Dinitrophenylhydrazine, Cu(II) or Fe(III) Schiff Base Complex, Antibacterial Activity.

INTRODUCTION

Schiff bases are a class of organic compounds formed by the condensation reaction between a primary amine and an aldehyde or ketone. This reaction leads to the formation of a characteristic imine (or azomethine) functional group (–C=N–), accompanied by the elimination of water. The term "Schiff base" is derived from the German chemist Hugo Schiff, who first reported these compounds in 1864.The general formula of a Schiff base is R_1–

CH=N–R_2, where:

- R_1 is usually an aryl or alkyl group derived from the carbonyl compound.
- R_2 originates from the amine group.

The lone pair of electrons on the nitrogen atom in the imine group imparts excellent ligand properties to Schiff bases, enabling them to coordinate effectively with metal ions. Significance of Schiff Base Ligands in Coordination Chemistry Schiff base ligands have gained significant attention in coordination chemistry due to several beneficial properties: Simple synthesis and easy structural modification, using a wide variety of aldehyde and amine precursors. Flexibility in donor atoms (e.g., N, O, S), enabling the formation of mono-, bi-, or multidentate complexes. Stability of chelate complexes with transition metals, improving chemical and biological behavior. and Catalytic Properties of Metal–Schiff Base Complexes Many metal complexes of Schiff bases demonstrate enhanced biological and catalytic activities, such as:

- Antibacterial, antifungal, and antiviral effects
- Catalytic performance in organic transformations
- Antioxidant and anticancer properties

Notable Metal Interactions-Transition metals like copper (Cu) and iron (Fe) form particularly effective Schiff base complexes. These interactions are essential due to their redox behavior and biological relevance. Schiff base ligands help stabilize these metal ions in unusual oxidation states and facilitate selective binding to biological targets.

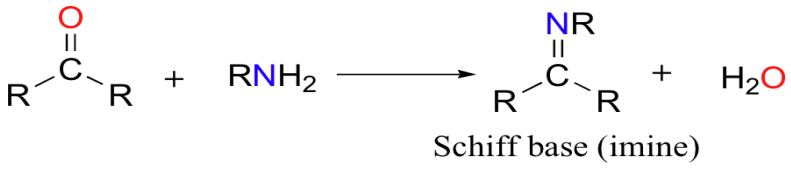


Fig. 1: General route for the preparation of Schiff bases.

MATERIALS AND METHODS

All reagents used were of analytical grade. The Schiff base ligand N-(1-(2,4-dinitrophenyl)ethylidene)hydrazine was synthesized by reacting 2,4-dinitrophenylhydrazine with acetaldehyde in ethanol. Metal complexes were prepared by reacting the ligand with copper acetate and ferric chloride. Characterization techniques included FTIR and UV-Vis spectroscopy. Melting point was determined using an electrothermal apparatus, and solubility was tested in various polar and non-polar solvents.

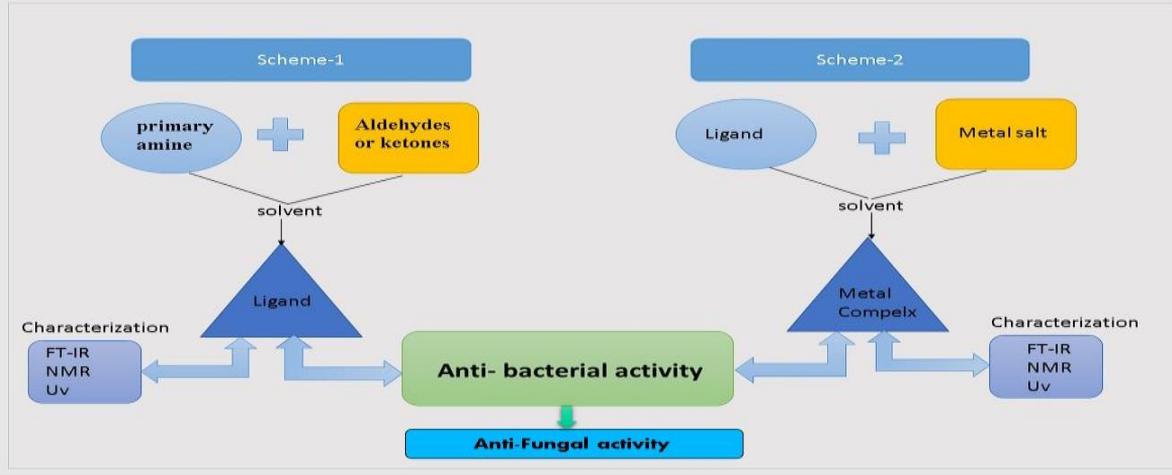


Fig. 2: graphical Abstract.

EXPERIMENTAL PROCEDURE

1. Synthesis of SCHIFF BASE LIGAND:- N-(1-(2,4Dinitrophenyl)ethylidene) hydrazine

Preparation of Schiff base:

1. Prepare 2,4-Dinitrophenylhydrazine solution: Dissolve 1g 2,4-Dinitrophenylhydrazine solution in 45mL ethanol.
2. Prepare Acetaldehyde solution: Dissolve 5mL Acetaldehyde in 5mL ethanol.
3. Combine solutions: Add Acetaldehyde solution dropwise to 2,4-Dinitrophenylhydrazine solution, stir at 45°C for 30min or until the mixture turns yellow.
4. Filter precipitates: Use filter paper or Buchner funnel.
5. Wash and dry: Wash with cold ethanol, dry to obtain product.
6. Recrystallize: Use hot ethanol to obtain pure crystals.
7. The final product should be N-(1-(2,4-Dinitrophenyl)ethylidene)hydrazine, a yellow crystalline solid.

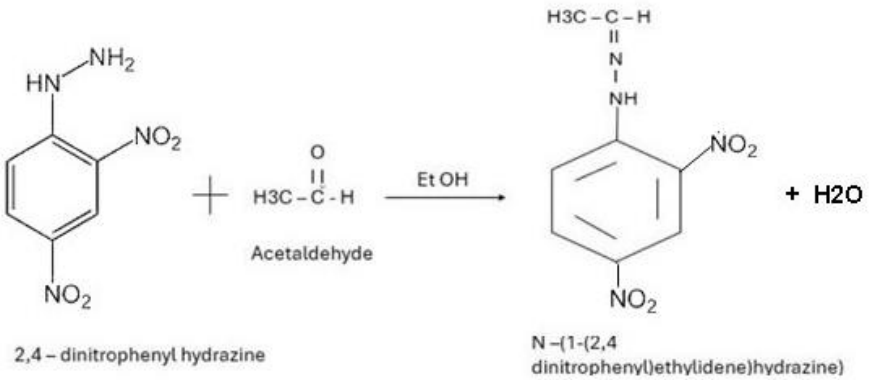


Fig. 3 General Route of Schiff-Base-Ligand.

SYNTHESIS OF FE (III) AND CU (II) COMPLEXES WITH SYNTHESIZED SCHIFF BASES

1. Synthesis Of Fe (III)Complexes With Synthesized Schiff Bases

STEPS FOR SYNTHESIS OF IRON(III) COMPLEX

1. Prepare N-(1-(2,4-Dinitrophenyl)ethylidene)hydrazine solution: 0.25g in ethanol.
2. Prepare Iron(III) Chloride solution: 0.34g in distilled water.
3. Combine solutions: Stir at 45°C for 30min.
4. Filter precipitates: Use filter paper or Buchner funnel.
5. Wash and dry: Wash with cold ethanol, dry to obtain pure Fe(III) complex.
6. **Bis(N-(1-(2,4-dinitrophenyl)ethylidene)hydrazine)Fe(III) chloride**

2. Synthesis Of Cu (II) Complexes With Synthesized Schiff Base

STEPS FOR SYNTHESIS OF COPPER COMPLEX

1. Prepare N-(1-(2,4-Dinitrophenyl)ethylidene)hydrazine solution: Dissolve 0.25g N(1-(2,4- Dinitrophenyl)ethylidene)hydrazine in ethanol.
2. Prepare Copper Acetate solution: dissolve 0.38g Copper Acetate in distilled water.
3. Combine solutions: Stir at 45°C for 30min or until green color appears.
4. Filter precipitates: Use filter paper or Buchner funnel.
5. Wash and dry: Wash with cold ethanol, dry to obtain pure green-colored product
6. **Bis(N-(1-(2,4-dinitrophenyl)ethylidene)hydrazine)copper(II)**

CHARACTERIZATION OF SYNTHESIZED LIGANDS AND THEIR COMPLEXES

Characterization of the ligands and its two metal complex Fe(III) and Cu(II) complexes are conducted by employing physical methods like color, solubility at different solvents, melting point, also spectroscopic methods namely FT-IR, UV-Vis and NMR spectrophotometers & Anti Microbial activity.

1. PHYSICAL CHARACTERISTICS:
2. UV-VIS SPECTROSCOPY:
3. FTIR SPECTROSCOPY:
4. NMR
5. ANTI-MICROBIAL ACTIVITY:

RESULTS AND DISCUSSION

The Schiff base ligand was obtained as a yellow crystalline solid. Cu(II) and Fe(III)

complexes were green and dark brown solids, respectively. FTIR spectra showed a shift in the C=N stretching band, confirming complexation. UV-Vis analysis revealed absorbance maxima at 330 nm for Cu(II) and 350 nm for Fe(III) complexes. The solubility tests indicated that the ligands and complexes were highly soluble in polar solvents like ethyl acetate and acetone, partially soluble in methanol and ethanol, and insoluble in water and n-hexane. Antibacterial testing showed that the Cu(II) complex had the highest zone of inhibition against E. coli, outperforming both the Fe(III) complex and the free ligand.

1. PHYSICAL CHARACTERISTICS OF THE SYNTHESIZED COMPOUNDS

A. Solubility test

The synthesized ligands and complexes were tested for their solubility in various solvents with a range of polarity like water, ethyl acetate, methanol, ethanol, acetone, and n-hexane. The synthesized ligands and complexes are highly soluble in polar solvents like ethyl acetate and acetone, partially soluble in methanol and ethanol but insoluble in water and n- hexane as shown in Table.

Note: X: Insoluble*: Partially soluble √:

Table 1: Solubility Of Ligands And Complexes.

Compounds	Solvents					
	Water	Ethyl acetate	Methanol	Ethanol	Acetone	n-hexane
Ligands	X	√	*	*	√	X
Cu(II) complex	X	√	*	*	√	X
Fe(II) complex	X	√	*	*	√	X

B. Melting point

Melting point measurements of synthesized ligands and complexes using an electro thermal melting point apparatus. The physical parameters like melting point, colour, of the synthesized ligands and their metal complexes.

Table 2: Physical properties of the synthesized compounds.

Compounds	Mol. formula	Mol. wt.	m.p°C	Color
Ligands	$C_8H_8N_4O_4$	224.14 g/mol	162 °C	yellow crystalline solid
Fe(II) complex	$C_{16}H_{16}ClFeN_8O_8$	539.67 g/mol	178°C	Yellowish –brown solid
Cu(II) complex	$C_{16}H_{16}CuN_8O_8$	511.92 g/mol	175°C	green solid

2. UV-VIS SPECTROSCOPY

1. UV Spectrum of synthesized Schiff Base ligand.

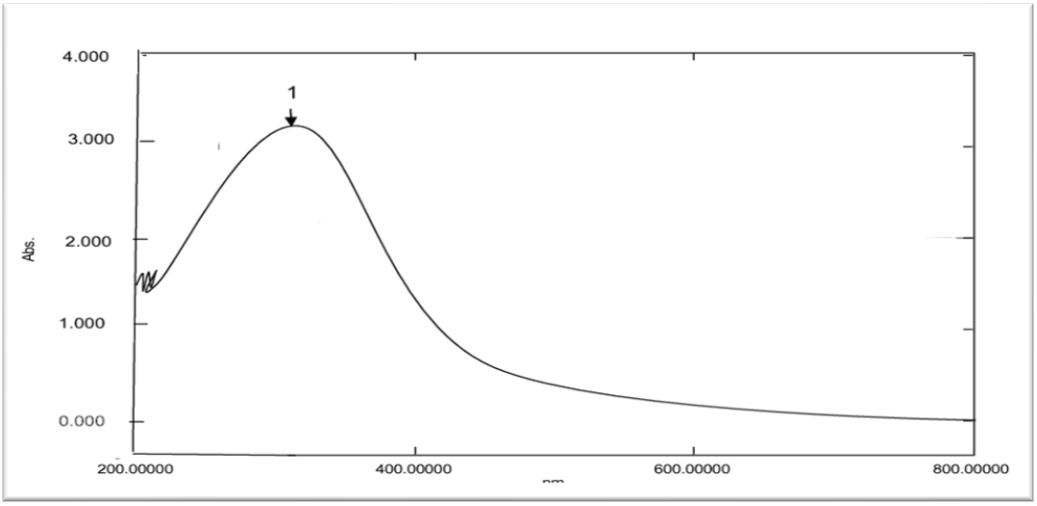


Fig. 4: UV Spectrum of Schiff Base.

The Schiff base ligands (**N-(1-(2,4-Dinitrophenyl)ethylidene)hydrazine,)** showed maximum absorbance of 1.534 at 350nm.

2. UV Spectrum of Copper(II) Complex

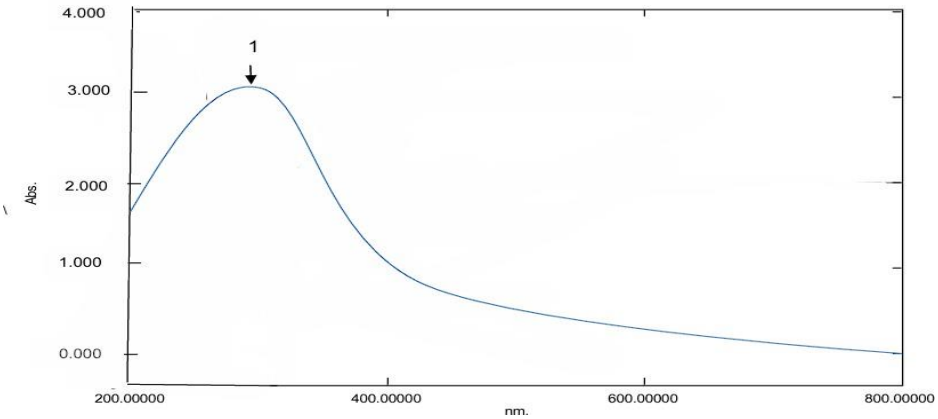


Fig. 5: UV Spectrum of Copper(II) Complex.

The metal complex of copper showed maximum absorbance of 1.850 at 330nm.

3. UV Spectrum of Fe(III) Complex

The metal complex of copper showed maximum absorbance of 1.873 at 350nm.

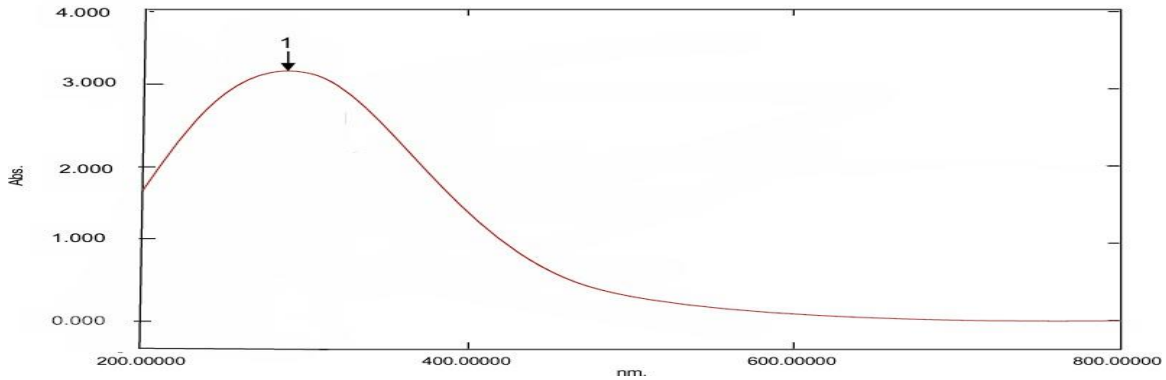


Fig. 6: UV Spectrum of Fe(III) Complex.

3. FTIR SPECTROSCOPY

There are some functional groups that can be distinguished by absorption using the IR spectrum. One of the most important functional groups that can be distinguished in compounds.

1. IR SPECTRUM OF SCHIFF BASE

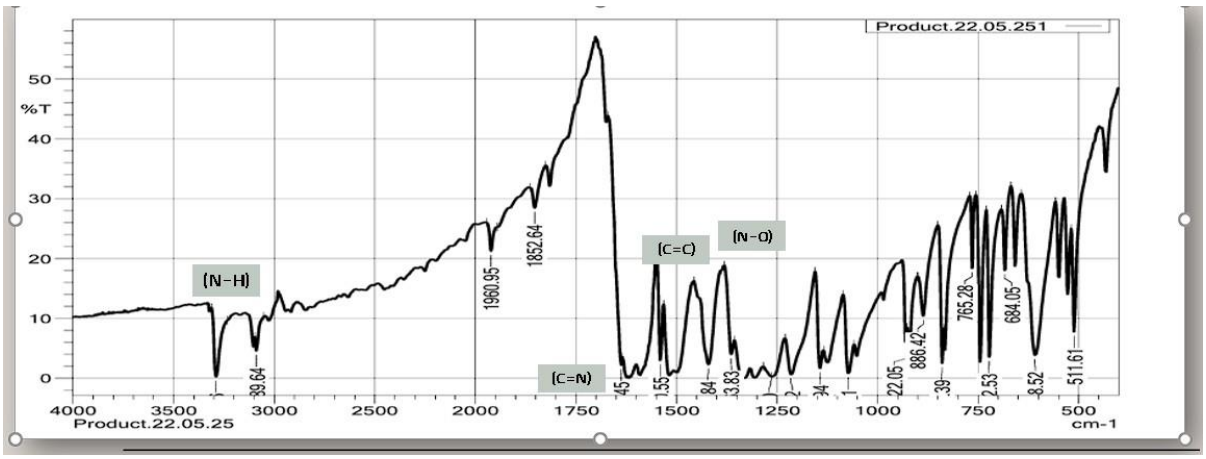


Fig. 7: Ir Spectrum of Schiff Base.

(C=C)	(C=N)	(N-H)	(N-O)
1540.55	1637.45	3289.16	1363.83

Standard range

(C=C) 1600-1500 cm-1 , (C=C) 1600-1500 cm-1, (N-O) 1500-1350 cm-1 (N-O) 1500-1350 cm-1 , (N- O) 1500-1350 cm-1

2. IR SPECTRUM OF FE(II) COMPLEX

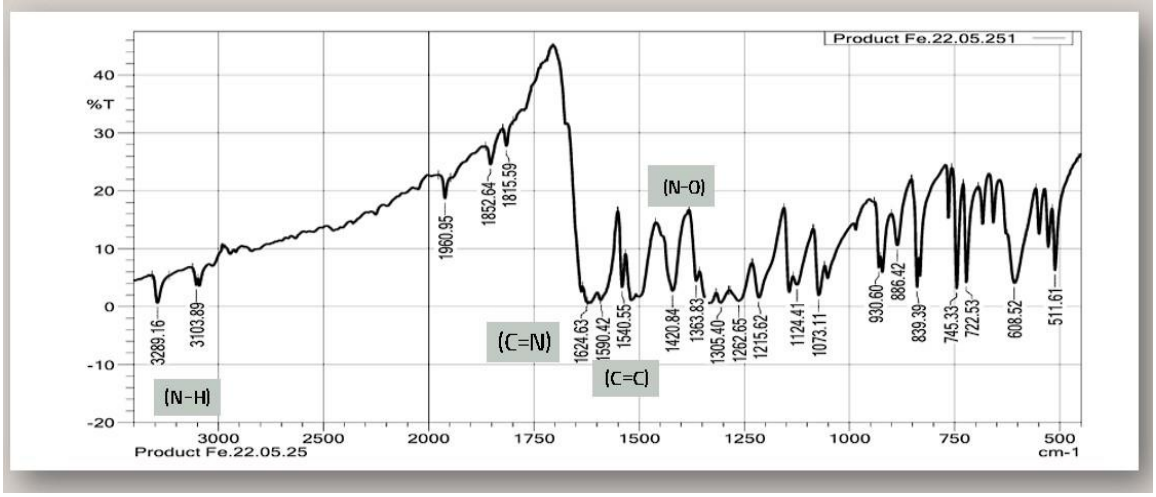


Fig. 8: Ir spectrum of fe(ii) complex.

(C=C)	(C=N)	(N-H)	(N-O)
1540.55 1590.42	1624.63	3103.89 3289.16	1305.40 1363.83 1420.84

Standard range-(C=C) 1600-1500 cm-1(C=C) 1600-1500 cm-1(N-O) 1500-1350 cm-1(N-O) 1500

1350 cm-1(N-O) 1500-1350 cm

3. IR SPECTRUM OF COPPER(II) COMPLEX

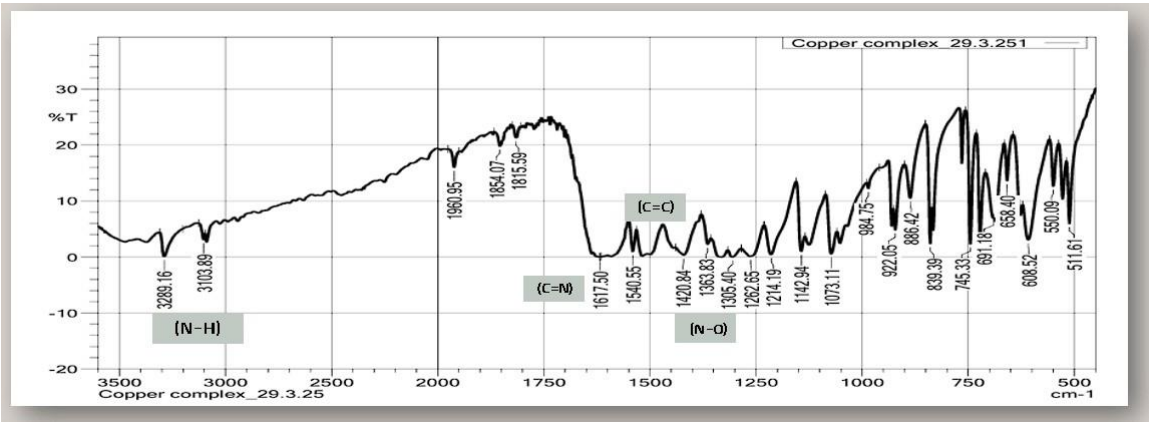


Fig. 9: Ir spectrum of copper(ii) complex.

(C=C)	(C=N)	(N-H)	(N-O)
1540.55	1617.50	3103.89 3289.16	1363.83 1420.84

Standard range

(C=C) 1600-1500 cm-1, (C=C) 1600-1500 cm-1, (N-O) 1500-1350 cm-1 (N-O) 1500-1350 cm-1, (N- O) 1500-1350 cm

4. NMR: Nuclear Magnetic Resonance

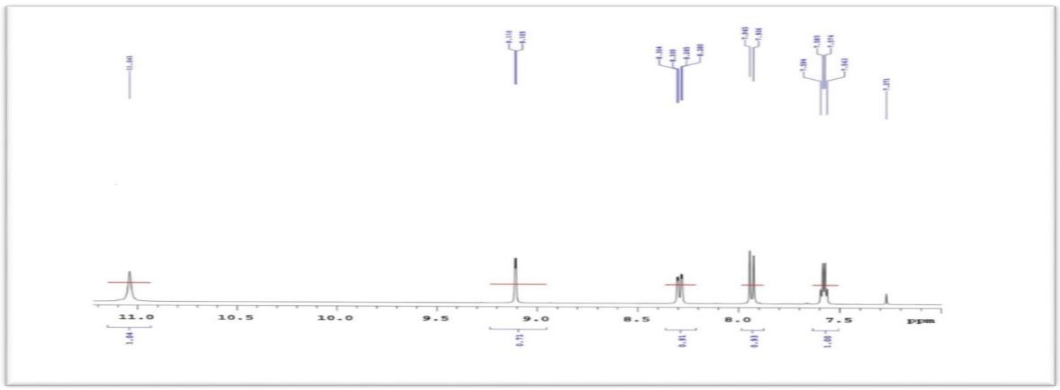
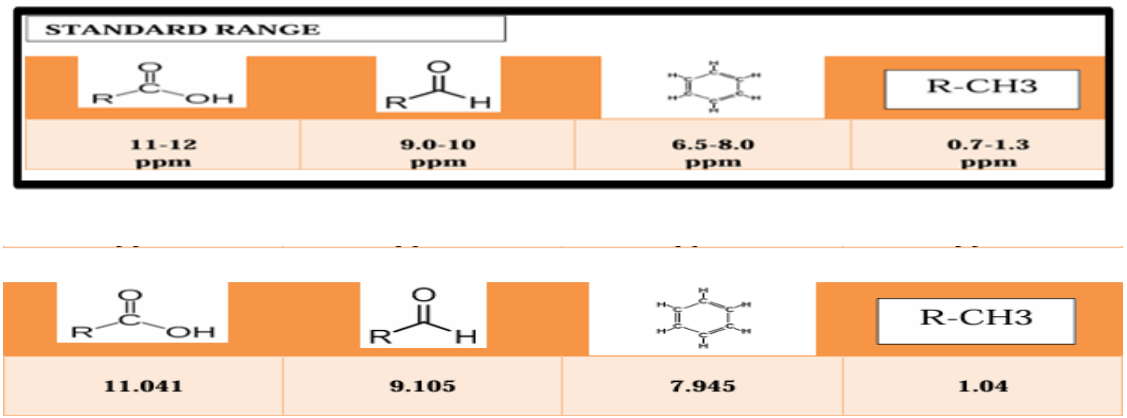


Fig. 9: NMR chemical shift of Schiff base ligands.



ANTI-MICROBIAL ACTIVITY

The in vitro anti-bacterial activity comparative study of the growth inhibition zone values of Schiff bases and their complexes against the Escherichia coli using disc diffusion method .standard –streptomycin.

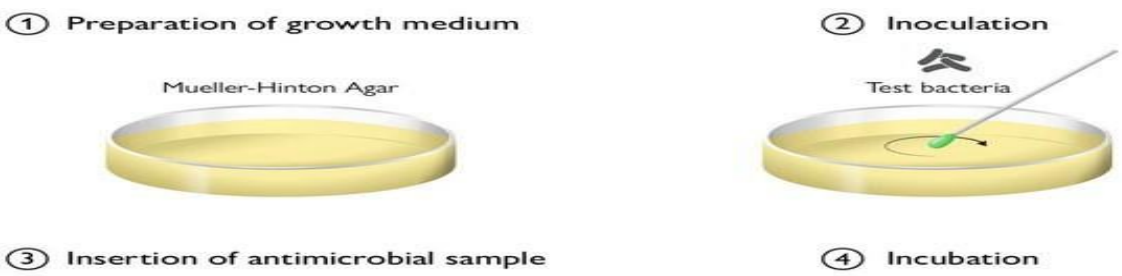


Fig. 10: In vitro anti-bacterial activity.

The antibacterial activity of Schiff base and their Cu and Fe metal complexes was evaluated against E. coli to assess their ability to inhibit microbial growth using streptomycin as a standard. The inhibition zones of antibacterial activity are presented in the Table

Table 3: inhibition zones of antibacterial activity.

compound	Zone of inhibition E. coli Radius (mm)
Streptomycin (standard)	20
Schiff Base ligands	13
Copper(II) Complex	15
Fe(II) Complex	10

The results of the antibacterial activity show that the Cu(II) complexes of ligand have showed excellent antibacterial activities in general, the results indicate that Cu(II) complexes gave highest activity or effective against the bacteria than the Fe(III) metal complexes and Schiff base ligand. However, The antibacterial activities of the synthesized Schiff base and its complexes exhibited moderate to good efficacy, although they were less potent compared to standard drugs."

CONCLUSION

Schiff base ligand n-(1-(2,4-dinitrophenyl)ethylidene)hydrazine and its Cu(ii) and Fe(III) complexes were successfully synthesized Structural characterization was confirmed through physical properties, FTIR, and UV-VIS spectroscopy, indicating successful metal coordination. Solubility tests showed better solubility of the compounds in polar organic solvents. Antibacterial studies revealed that the Cu(ii) complex exhibited the highest activity against E.Coli, followed by the Fe(III) complex and the ligand.

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