

Synthesis and antibacterial activity of 3-nitrobenzaldehyde semicarbazone ligand and its Ni (II) and Cu (II) Complexes

Abstract

Schiff bases are the most widely used organic compounds. They have been shown to exhibit a broad range of biological activities, including; antifungal, antibacterial, anti-malarial, anti-proliferative, anti-inflammatory, antiviral, and antipyretic properties. In this study semicarbazone ligand was prepared by condensing 3-nitrobenzaldehyde with semicarbazide hydrochloride in 1:1 molar ratio in ethanolic medium. This ligand was used to synthesize metal complexes of copper (II) and nickel (II) in 1:2 molar ratio using ethanol as a solvent. Characterization and structure elucidation of prepared metal complexes have been investigated on the basis of molar conductance and UV and IR spectral studies. The all prepared compounds showed a vital effect against both types of bacteria gram positive (*Staphylococcus-aureus*) and gram negative (*Escherichia-coli*).

Keywords: schiff bases, metal complexes, antibacterial activities

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Introduction

Schiff bases play important role in co-ordination chemistry as they easily form stable complexes with most transition metal ions. Many biologically important Schiff bases and their metal complexes have been reported in literature possessing, analytical, industrial, biological, clinical, biochemical, antimicrobial, anticancer, antibacterial, antifungal and antitumor activity^{1,2} in addition with important roles in ranging from anticorrosion, soil treatment agents and medicinal agents.^{3,4} Schiff base ligands and their transition metal complexes have been extensively investigated due to their wide range of applications including catalysts, medicine, crystal engineering, anticorrosion agent.^{5,6} Schiff bases are widely studied due to their synthetic flexibility, selectivity and sensitivity towards the central metal atom, structural similarities with natural biological compounds and also due to presence of azomethine group (-N=CH-) which imports in elucidating the mechanism of transformation and racemization reaction biologically. Schiff bases having chelation with oxygen, nitrogen etc. Imine or azomethine groups are present in various natural, derived and non natural compounds. The imine group present in such compounds has been shown to be critical to their biological activities.⁷ Schiff base complexes containing two or more metal centres are efficient catalysts. It is also well known that coordination of a ligand to metal ion acts synergistically to increase the biological activity of the ligand and decreases the cytotoxic effects of metal ion and ligand.⁸ This study concentrates on the synthesis and biological activity of Schiff base and its metal complexes.

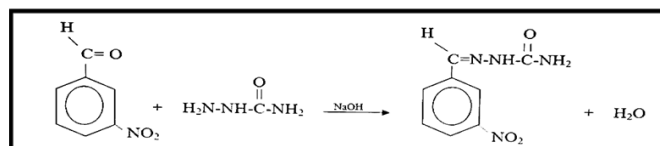
Materials and methods

Chemicals and reagents

Semicarbazidehydrochloride, Nickel chloride, Copper chloride dihydrate, Sodiumhydroxide (CDH), 3-nitrobenzaldehyde (FSA), Absolute ethanol (GCC).

Synthesis of ligand

(0.01mol, 1.11g) of semicarbazide hydrochloride and (0.01mol, 0.4g) of Sodium hydroxide were dissolve in (20ml) of a hot ethanol and (0.01mol, 1.51g) of 3-nitro benzaldehyde was dissolve in (20ml) of a hot ethanol and shake well with constant stirring at 78°C for 7hrs. Yellow precipitate separated out filtered and dried Scheme 1.



Scheme 1 Synthesis of 3-nitrobenzaldehydesemicarbazone Ligand.

Synthesis of complexes

Substituted ligand (0.0002mol, 0.042g) was dissolved in (20ml) of ethanol and (0.0001mol) of metal salts Cu(II) or Ni(II) in (20ml) of warm ethanol was added to the previous solution with stirring for 3hrs. The reaction mixture was allowed to stand and then filtered and dried. Reaction scheme was shown Figure 1.

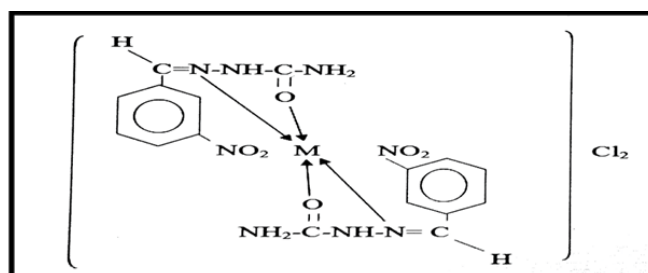


Figure 1 Proposed structures of the Cu (II), Ni (II) complexes.

IR spectral analysis

The appropriate weight of the ligand and their complexes was record and grinding with potassium bromide to form disk out let for the infrared (IR) spectrum record within the range 500-4000cm⁻¹.

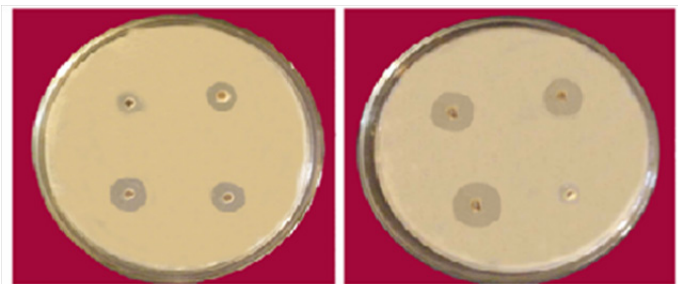


Figure 2 Antibacterial activity of Semicarbazone Cu (II) and Ni (II) complexes against *E. coli*.

UV-Vis spectral analysis

A solutions with a concentration of (1×10⁻³ mol.L⁻¹) for the ligand and (1×10⁻⁵ mol.L⁻¹) for the complexes were prepared. The absorbance spectra were recorded in the visible and ultraviolet region with 200-800 nm using ethanol as a reference solution.

Conductivity measurement

Attended solutions concentration of (2×10⁻³ mol.L⁻¹) for the prepared complexes and measured the electrical conductivity.

Antimicrobial activity

Preparation of nutritious medium for the growth of bacteria

Take 28 grams of nutrient agar and dissolve in 1000 ml of

distilled water, then set at the water bath at 45°C until the solution is homogenized and sterilized in autoclave at 121°C for 15-20 minutes Press 15 psi and leave the solution to cool down to 47°C and then pour in Petri dishes.

Preparation of the blood nourishing medium for bacterial growth

After leaving the solution to the blood stream.

Method of working fossils known diameters

The craters worked on the surface of the nutrient medium in Petri dishes, which grew in the bacterial colonies and the hole diameter of 8 mm with a hole in the center of the dish filled with absolute alcohol for methanol or distilled water for control. Measurements were made for the diameters of the extract, which were poured into holes through the diaphragm (Verna). The calculations were carried out to determine the extent of the spread of the extract on the bacterial colonies, “diameter of the extract-diameter of the hole” and the results were recorded between the ligand and metal ion. The complexes were dissolved in ethanol and the results showed that the complexes exhibit high melting points, indicating a strong bonding the height conductance values of the complexes support their electrolytic nature of the compounds to cool down to 47°C, add 2.5 ml fresh human blood and mix well, then pour on clean and sterile Petri dishes.

Results

Physical properties of ligand and its complexes

The ligand and its complexes are stables in air they have varied colors, and the all prepared compounds are soluble in ethanol. Melting points were measured using open capillary tubes on melting point apparatus and the molar conductivity of (2×10⁻³ mol.L⁻¹) of their solution was measured at room temperature (Table 1).

Table 1 Physical properties of ligand and its complexes

Compound	M. wt	Colour	Appearance	Yield %	m.p°C	Molar Conductance (Ω ⁻¹ cm ² mol ⁻¹)
L ₁	208	Yellowish	Powder	73.5	249	-
[Cu(L) ₂]Cl ₂	549	Yellow	Crystal	57.6	252	119
[Ni(L) ₂]Cl ₂	544	Orange	Crystal	50	265	147.8

L=C₈H₈O₃N₄, M.wt=Molecular weight, m.p=melting point

Electronic spectral data of ligand and its complexes

Electronic spectra of complexes have band at (312-313 nm) assignable to π-π*.⁹

IR data of ligand and its complexes

The IR spectrum of the prepared compounds showed absorption peaks at 1683-1685cm⁻¹ due to the stretching of the conjugate C=N¹⁰ and the appearance of absorption peak at 1716cm⁻¹ due to the expansion of the association C=O and note that the decrease of this absorption in the complex, indicating the metal bonding oxygen¹¹ and the appearance of absorption peak at 3161cm⁻¹ due to NH the reduction of the absorption in the complex, indicating the association

of the metal with the nitrogen and the emergence of absorption in the region 3464cm⁻¹ is due to the NH₂ group¹² and the appearance of absorption peaks in the region 522-630cm⁻¹ and 451-563cm⁻¹ due to the metal bonding with oxygen and nitrogen respectively (table 2).¹³

Antibacterial activity

The synthesized ligand and its corresponding complexes were screened for their antibacterial activity against two types of bacteria Gram-negative (*Escherichia-coli*) and Gram-positive (*Staphylococcus-aureus*). As shown in table 3 the activity determined by using the Inhibition zone technique. All tests the organisms were growth on nutrient agar medium. Methanol was used as standard reference compound.

Table 2 IR and UV-Vis spectral data of ligand and its complexes

Compound	$\nu(\text{NH}_2)$	$\nu(\text{NH})$	$\nu(\text{C=N})$	(C=O)	$\nu(\text{M-O})$	$\nu(\text{M-N})$
L ₁	3464 _m	3161 _m	1683 _m	1716 _s	-	-
[Cu(L ₁) ₂]Cl ₂	3464 _m	3159 _m	1685 _m	1712 _s	630 _w	563 _w
[Ni(L ₁) ₂]Cl ₂	3464 _m	3159 _m	1683 _m	1712 _s	522 _w	451 _w

L= ligand, m = medium, s = strong, w = weak

Table 3 Antibacterial activity of ligand and its metal complexes

Compound	Concentration	Diameter of inhibition zone (mm)	
		<i>Escherichia-coli</i>	<i>Staphylococcus-aurous</i>
Ligand (L)	(2×10 ⁻³ mol.L)	9	8
[Cu(L) ₂]Cl ₂	(2×10 ⁻³ mol.L)	13	10
[Ni(L) ₂]Cl ₂	(2×10 ⁻³ mol.L)	16	13

Conclusion

In This study we have synthesized biologically active semicarbazone ligand and it's Cu (II) and Ni (II) complexes. The synthesized ligands and their derivatives were characterized and identified on the basis of physical and spectral data. Antibacterial activities were found that metal complexes are more active than the ligands that are indicated that the coordination increases their bioactivity. The more investigations are going on with this hope that some of these compounds may be used as antimicrobial agent.

Acknowledgments

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Conflicts of interest

Authors declare that there is no conflict of interest.

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