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Research Article

**Synthesis and Characterization of Schiff Base of
p - chloro aniline and their Metal Complexes and their
evaluation for Antibacterial Activity**

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Abstract

Properties of schiff bases and their metal complexes have been identified by various scientists in the past and also lots and lots of work is also going on to identify the unique properties of these schiff bases. Here another attempt was made to identify the properties of *p*-chloro aniline schiff base and its derivative. Major focus was given on antimicrobial activity of schiff base and its metal complexes. Chemical properties were determined using UV Vis spectrophotometer, IR spectrophotometer and CHN analyzer.

Keywords : *p*-chloro-aniline, schiff base, metal complex of schiff base, antimicrobial activity,

INTRODUCTION

A Schiff base (or azomethine), named after Hugo Schiff, is a functional group that contains a carbon-nitrogen double bond with the nitrogen atom connected to an aryl or alkyl group—but not hydrogen. i.e. $R_1R_2C=N-R_3$, where R_1 is an aryl group, R_2 is a hydrogen atom and R_3 is either an alkyl or aryl group. The nature of the R group is limited to alkyl or aryl substituent's or hydrogen at the point of attachment to the imino (C=N) carbon or nitrogen.¹⁻³

The nomenclature for compounds of this type is often variable. Some chemical abstracts covered these materials under the categories imines and Schiff base. For convenience, it is felt that the term Schiff base should be limited to designating only those imines where R is an aryl group, R' is a hydrogen and R'' either an alkyl or aryl group.^{1,2}

Schiff bases are formed by condensation reaction. A number of reviews on Schiff base synthesis are available. Schiff base compounds are commonly

referred to by their names abbreviations. The abbreviations are a combination of the ketones and amine precursors. For example, bis(acetyl acetone) ethylenediimine becomes acacen, and N.N'-bis(Salicylidene) ethylene diamine becomes salen. Schiff bases could be bidentate, tridentate, tetra dentate or poly-dentate ligand capable of forming very stable complexes with transition metals. They can only act as coordinating ligand if they bear a functional group, usually the hydroxyl, sufficiently near the site of condensation in such a way that a five or six member ring can be formed when reacting with a metal ion. Schiff bases derived from aromatic amines and aromatic aldehydes have a wide variety of applications in many fields, e.g. biological, inorganic and analytical chemistry. Applications of many new analytical devices require the presence of organic reagents as essential compounds of the measuring system.^{1,3,4}

Schiff bases are used in various sensors mostly in optical and electrochemical as well as in certain chromatographic techniques, to detect the enhanced selectivity and sensitivity^{5,6}. It is also found that, certain schiff bases possess very good characteristics and structural similarities with natural compounds. Simple preparation methods of schiff base and the synthetic production efficiency enable to design the specific structure. Schiff bases of primary amines as well as carbonyl compounds are highly used in analytical determination. They also play an important role in coordination chemistry due to their ability to make stable complexes with most transition metal ions. In organic synthesis, Schiff base reactions are mostly used in making carbon-nitrogen bonds.⁵

Schiff bases are also found to play an important role in certain enzymatic reactions, which involve interaction of the amino group of the enzyme, with a carbonyl group of the substrate. Stereo chemical investigations carried out with the aid of molecular models showed that Schiff bases formed between methyl glyoxal and the amino group of the lysine side chains a Charge transfer can occur between these groups and the oxygen atoms of the Schiff bases. Schiff bases derived from pyridoxal (the active form of vitamin B6) and amino acids are considered as very important ligand from biological point of view.^{3,4}

Schiff bases are serving as intermediates in non-enzymatic glycosylation. These processes are very common normal during aging but could be accelerated in certain conditions caused by stress, excess of metal ions or diseases like Alzheimer's disease, diabetes, and atherosclerosis.⁷⁻⁹ There are numbers of biologically important Schiff bases have been noted in previous study possessing, antibacterial, antifungal, antimicrobial, anticonvulsant, antitumor, anti-inflammatory, and anti HIV activities. The other important role of Schiff base structure is in transamination. Transamination reactions are catalyzed by transaminases enzymes. They are present in mitochondria and cytosol cells. All the transaminases possess same prosthetic group, i.e., pyridoxal phosphate, this is covalently attached to schiff base via an imino group.^{8,9}

pyridoxal and amino acids derived schiff bases can act as very important ligands from the biological point of view. The faster development of these type of ligands leads to enhanced research activity in the field of coordination chemistry which is leading to very interesting conclusions. Not only these, many polymeric schiff bases have also shown antitumor activity.^{10,11} These schiff bases have optimum hydrolysis activity at pH 5 along with maximum water solubility. A complex has been defined as a

species formed by the association of two or much simpler species each capable of independent existence. When one of the simpler species is a metal ion, the resulting entity is known as a metal complex. There are certain metallo-elements without which the normal functioning of the living organism is inconceivable, among these metallo-elements so called, 'metals of life', four members form an 'island'. These are Na, Mg, K and Ca. Among the transition elements are V, Cr, Mn, Fe, Co, Ni, Cu and Zn. Na, Mg, K and Ca play vital roles in the living system and are present at trace and ultra trace quantities in the cell. These are also known as 'metals of life'. These transition elements are known to form Schiff base complexes.^{8,9}

In the present study schiff base of p-chloro aniline was synthesized and their metal complexes were prepared. These metal complexes were further characterized and their antimicrobial activity was also determined.

MATERIALS AND METHODS

Synthesis of p-chloro aniline N-salicylidene Schiff base

In the reaction, 1.1gm of salicyldehyde was mixed with 10 ml of ethanol, to this ethanolic solution 1.28g P-chloro aniline was added. The solution was mixed on magnetic stirrer with addition of 2 to 3 drops of concentrated H₂SO₄. Then it was refluxed for 2 h and left overnight at room temperature. The solid colored product was recovered by was filtration, which washed with ethanol and ether consecutively. It was dried at room temperature and recrystallized with hot ethanol to obtain Schiff base.

Preparation of the Schiff base Metal Complexes

TO start the reaction, 4.62 g (0.02mol) of Schiff base was dissolved in 25 ml of ethanol. To this solution, 0.01 mol metal solution was added which was dissolved in 20 ml of ethanol (1:2 metal-ligands). This mixture was magnetically stirred constantly and refluxed for 2 h. The obtained product was cooled to room temperature. Colour precipitates were collected by filtered and washed with ethanol and vacuum dried. (Three metal were used for the study: CuCl₂, CoCl₂, CdCl₂)

UV-VIS Spectrophotometer

Spectrum analysis was carried out by UV 1800 dual beam spectrophotometer, Shimadzu, Japan.

FTIR Analysis

FTIR analysis was done for confirmation of production formation. Shimadzu IR affinity-I along with DRS 8000A was used for the analysis.

Elemental Analysis

CHN analysis was done using Perkin Elmer, series-II, USA with accuracy 0.3% and analysis time 6-8minutes.

Antimicrobial Activity

Antimicrobial activity was carried out by agar cup method against *E.coli*, *S.aureus*, *Klebsiella* and *Pseudomonas*. Two different concentrations 50mg/L and 100mg/L were used for determination of the activity. Zone of inhibition was measured in mm.

RESULTS AND DISCUSSION

Physical Properties of Schiff base and their Metal complexes

Some of the physical properties of these schiff base and metal complexes were analyzed and recorded as mentioned in table 1. It was found that all the metal complexes have comparatively higher melting point than the schiff base. This indicated the higher stability of the compound after binding with metal. Similar kind of observation was made by Cinarli *et al* (2011) and Chowdhury *et al* (2010) in their studies.^{12,13}

Spectrum Analysis

In the spectrum analysis of schiff base of p-chloro aniline N-salicylidene optimum absorption was measured at 237 nm. While in case of metal complexes red shift in the spectra was observed. Co has given optimum absorption at 375 nm, Cd at 380 nm and Cu at 370 nm.

FTIR Analysis

I.R. spectra of the Schiff base (Figure 1) showed the absence of bands at ~1735 and 3315 cm⁻¹ due to the

carbonyl $\nu(\text{C}=\text{O})$ and $\nu(\text{NH}_2)$ stretching vibrations and a strong new band appeared at ~1630 cm⁻¹ assigned to azomethene $\nu(\text{HC}=\text{N})$ linkage, showing that amino and aldehydes moieties of the starting material are absent and have been converted into the ligand, i.e. p-chloro aniline N-salicylidene.

Elemental Analysis

Elemental analysis has confirmed the incorporation of metal in the schiff base. The following table shows the percentage of metal incorporation in the schiff base (table 2).

Antimicrobial Activity

Table 3 shows the antimicrobial activity of the schiff base and its metal complexes. It was found that metal complexes are more effective in inhibiting the microbial growth. Similar kinds of observation were made in many previous studies.^{14,15} The possible reason behind this may be the interaction of the metal complexes with lipoproteins of the cell. These metal complexes may not allow the normal functioning of the microbial cell.^{6,16,17} Not only this, their higher stability at higher temperature may also allow them to use them as a potential antimicrobial agent.

CONCLUSION

When the Schiff base and its metal chelates were evaluated for their antibacterial activity. It was observed that the metal chelates are more effective antimicrobial than the respective uncomplexed ligands. This concludes that certain processes of chelation dominantly affect the biological behavior of the compounds with higher potential against some bacterial strains.

Table 1
Physical properties of Schiff Base and Metal complexes

No.	Color	Solubility	Melting Point °C
L1	Yellow	Ethanol, DMF, Acetone	123
Co	Bright Yellow	Ether, DMF, DMSO, Methanol	>152
Cu	Pale yellow	Ether, DMF, DMSO, Methanol	>152
Cd	Yellow	Ether, DMF, DMSO, Methanol	>155

L1-Schiff base, Co-Cobalt Complex, Cu-Copper Complex, Cd – Cadmium Complex

Table 2
Elemental Analysis

Compound	% found/calculated			
	% C	% H	% N	% Metal
L ₁	71.36(75.40)	6.08(7.35)	7.04(7.96)	-----
Co	52.40(59.90)	4.24(5.80)	7.23(6.35)	12.23
Cu	53.31(54.30)	5.07(6.26)	4.11(4.87)	12.39
Cd	47.58(43.54)	5.65(4.94)	5.09(4.61)	12.15

L₁-Schiff base, Co-Cobalt Complex, Cu-Copper Complex, Cd – Cadmium Complex

Table 3
Antimicrobial activity of schiff base and metal complexes

Schiff base	Zone of Inhibition in mm							
	<i>E.coli</i>		<i>S.aureus</i>		<i>Klebsiella</i>		<i>Psuedomonas</i>	
	50 mg/L	100 mg/L	50 mg/L	100 mg/L	50 mg/L	100 mg/L	50 mg/L	100 mg/L
L ₁	14	16	14	15	12	14	12	13
Cd metal	17	19	19	20	18	21	18	20
Cu metal	18	22	18	21	17	18	17	19
Co metal	18	22	22	23	18	20	20	22

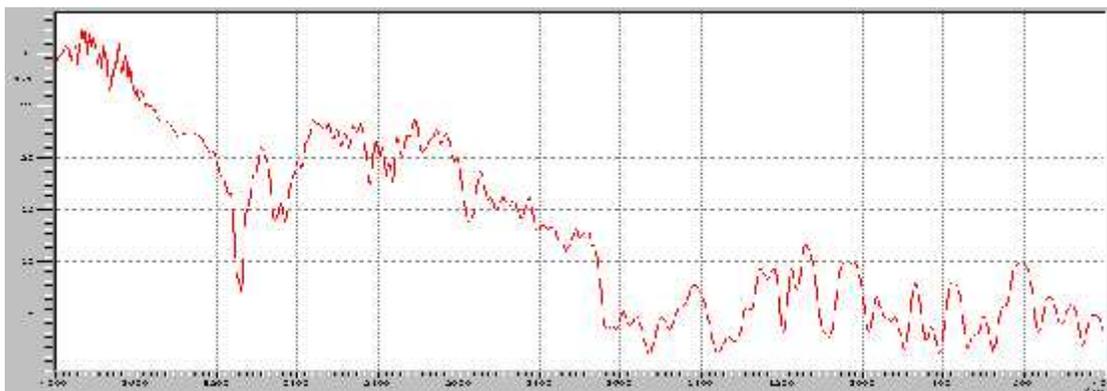


Figure 1
FTIR analysis

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