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Research Article

Synthesis, Spectroscopy and Biological Studies of Copper and Silver Complexes Derived from 2-Substituted Amino Propanoic Acid

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Abstract

Synthesis of copper and silver complexes were prepared from novel Schiff base ligand. These synthesized ligand and their metal complexes were characterized by elemental analysis, FT-IR, ^{1}H NMR, UV-Vis and Mass spectrometry. The coordination of amino nitrogen, phenolic oxygen, and carboxylato oxygen atoms is shown by the infrared spectra.The Schiff bases and its complexes were screened for their in vitro antibacterial and antifungal studies against the bacteria Pseudomonas Aeruginosa (PA, Gram -ve), Bacillus Cirroflagellosus (BC, Gram +ve), Penicillium Notatium (PN) and Aspergillus Niger (AN) strains. The results of the antibacterial investigation show that the copper and silver complexes possess more potent bactericide and fungicide properties than the ligand.

Keywords: Transition metal complexes, UV-Vis, FT-IR studies, antibacterial studies, antifungal studies.

1. Introduction

The importance of synthesis and chemical properties of Schiff base transition metal complexes has increased due to extensive range of uses for many years [1-3]. By attaching two or more than two metal centres in close nearness, polydentate and compartmental ligands can create such multimetallic clusters [4]. The production of Schiff base intermediates in biologically [5] important processes is extensively known. However, the variables that regulate the creation of such intermediates have received less attention. It is observed that Cu (II) and Ag (II) complexes derived from Schiff base ligands of varying denticity [6]. The current study comprises the synthesis and characterisation of vanillin and D (+) alanine-containing copper (II) and silver (II) Schiff base complexes. The biological activities can also be improved by complexation with transition metal ions. A compound having heterocyclic ring system with both N and S in their structure shows excellent biologically active property [7]. Schiff base ligands are potential ligand due to the ease of preparation as well as single step condensation of primary amines and aldehydes in organic solvent such as alcohol [7,8]. Schiff bases are widely used in several fields, such as intermediates in dyes, organic synthesis, biological, catalysis, , polymer stabilizers and pigments [9,10]. The complexation of these ligands with metal are pervasive because of their potentiality for various chemical modifications, synthesis, and wide applications. Schiff bases and their metal complexes are reported to have a broad range of biological activities, such as antimalarial, antibacterial, antioxidant, antiviral and various other activity [11-15]. Various kinds of antibacterial agents have been disclosed in the past two decades [16]. Schiff base complexes posses biological interest and are utilized as almost successful models of biological compounds. The phenoxo oxygen atom (or ketone oxygen) and azomethine nitrogen atom present in these type of ligands shows good binding ability and thus are very effective in showing coordination behavior with central metals to form complexes [17-19]. In the present work, this field is extended in synthesis of Schiff base complexes, which can be used in numerous applications. The ligand is prepared by the

condensation of D (+)alanine with o-vanillin respectively, the ligand further treated with transition metal such as copper and silver salts to form metal-ligand complexes with appropriate amounts of their moiety [20]. The spectroscopic studies gave insights about the chelation and coordination mode of two different ligands with copper and silver.

2. Experimental

2.1. Material and methods used

The chemicals such as D (+)alanine, ortho-vanillin (2-Hydroxy-3-methoxybenzaldehyde), Cupric sulphate (pentahydrate) and silver nitrate($AgNO_3$) were purchased from Sigma Aldrich and used without purification. Prior to use, solvents were purified and dried in accordance with the accepted procedure [21]. Complexometric titrations using EDTA were used to determine the metal contents [22]. A Perkin-Elmer elemental analyser was used to acquire the elemental analyses (C, H, and N). A JASCO/FT-IR spectrometer was used to detent the infrared spectra of KBr pellets between 400 and 4000 cm^{-1}. Using ethanol as the solvent, electronic spectra in the 200-900 nm range were captured using a Perkin-Elmer LAMBDA25 UV/Vis spectrometer. Using a calibrated digital conductivity metre and freshly generated 10^{-3} M EtOH solutions, conductance measurements were executed at room temperature.

2.2. Synthesis

2.2.1. Synthesis of Ligand

D (+)alanine (0.58 g, 6.5 mmol), dissolved in EtOH (10 mL), was added slowly with constant stirring to an alcoholic solution (20 mL) containing KOH (0.36 g,6.5 mmol). After stirring for about 30 minutes, the solution was filtered, ortho-vanillin (1.0 g, 6.5 mmol) was added slowly dropwise while stirring continuously to the filtrate, which was dissolved in EtOH (20 mL) (Figure 1). The resulted solution obtained was yellowish which was evaporated under reduced pressure and kept at room temperature for three days. The precipitate was filtered, washed with cold alcohol and Et_2O [23]. It was recrystallised from methanol (Yield = 58%).

2.2.2. Synthesis of Transition metal complexes

A solution of ethanol (25 mL) and Schiff base (0.222 g, 1.0 mmol) was mixed with a solution of copper (II) sulphate and silver(II) nitrate (1.0 mmol) in EtOH and the resulted mixture was stirred at room temperature for an hour (Figure 2). The resulting mixture was cooled to room temperature and allowed to evaporate on a water bath at 50°C for about two hours. After filtering, the precipitate was washed with cold alcohol and water. The resultant precipitate was then dried in a vacuum desiccator after being recrystallized from MeOH [24] (Yield = 52%).

2.2.3. Antibacterial activity

To test the activities like antibacterial and antifungal activities of ligand, copper and silver complex the Agar plate method was used using Grisofluvin and Norfloxacin as standards, against the bacteria Pseudomonas aeruginosa (PA, Gram -ve), Bacillus Cirroflagellosus (BC, Gram +ve), Penicillium Notatium (PN) and Aspergillus niger (AN).The consequences of the antibacterial and antifungal review are given in Table 1. In ethanol, the substance tested had a concentration of 1 mg/mL. On the medium of agar, which had been inoculated with microorganisms, a well was made as usual.

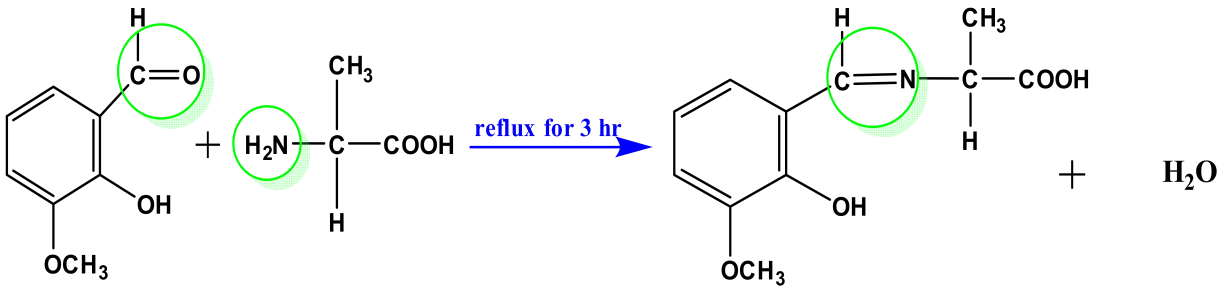

Figure 1. Synthesis of Schiff base ligand.

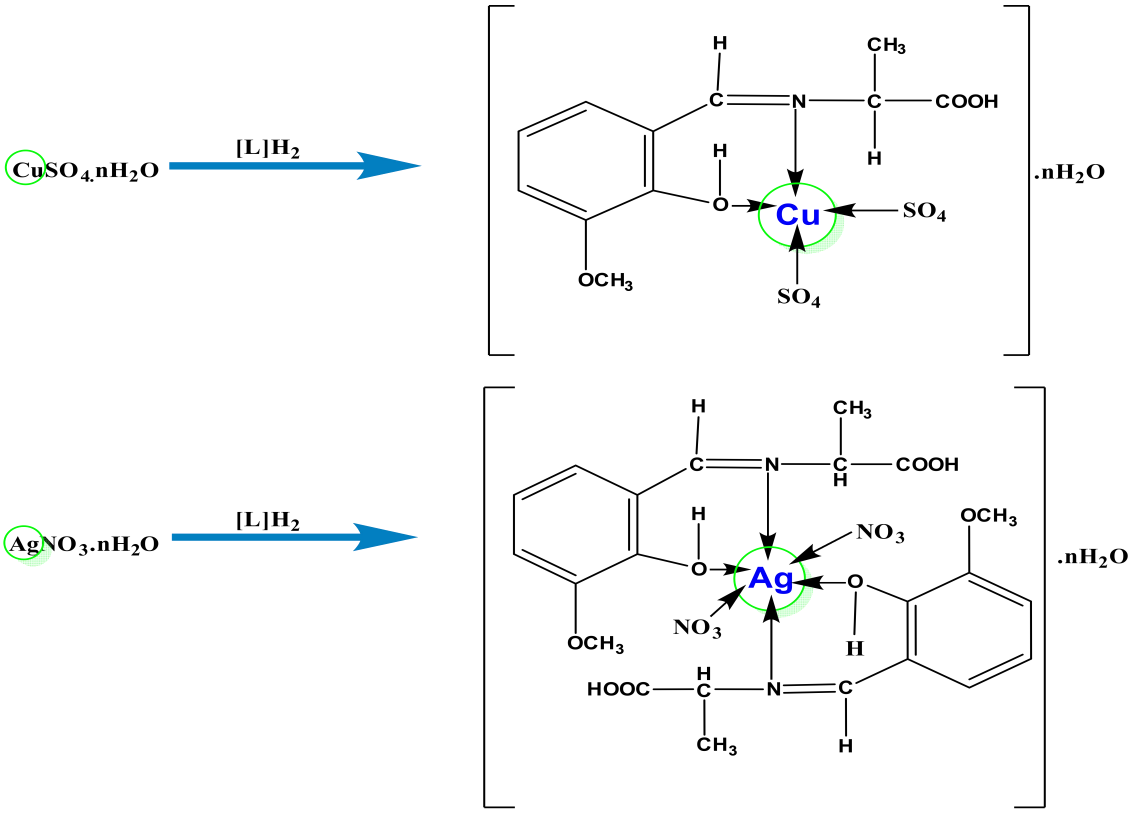

Figure 2. Synthesis of Copper and silver complexes

Table 1. Antibacterial and antifungal activity data of ligand and its complexes

Complexes	Antifungal PN	Antifungal AN	Antibacterial PA	Antibacterial BC
$[L]H_2$	-	-	+	+
[CuLX]	-	-	++	++
[AgLX]	+	+	++	++

Note: *(-) No inhibition zone=inactive; 1–5 mm (+) = less active; 6–10 mm(++) = moderately active; (PN = Penicillium Notatium, AN = Aspergillus Niger, PA=Pseudomonas Aeruginosa, BC = Bacillus Cirroflagellosus)

Using a micropipette, the test solution was poured into the well and the plate incubated at room temperature for 48 hours. During this time the test solution dispersed and affected the growth of various microorganisms.The size of the inhibition zone around the cups was used to estimate the antimicrobial activity [25, 26]. The ligand was less effective against PA and BC, Cu complex shows moderate activity against PA and BC, silver complex show less effectiveness against PN and AN but moderate activity against PA and BC.

3. Results

3.1. ^{1}H NMR spectra

The ^{1}H NMR spectra of the ligand show six signals (Table 2). By observing and comparing the ^{1}H NMR data of the copper and silver complex with those of the ligand, it is seen that the

proton signal for the amino (-CH=N-) proton at 11.12 ppm in the ligand is shifted upfield to 11.06 ppm in the copper complex and 7.26 ppm in the silver complex showing that the free ligand is coordinated with the copper ion [27, 28] and silver ion respectively.

3.2. Infrared Spectroscopy

The FT-IR spectra for the ligand and its metal complexes shown in Table 3. The comparison with the vibrational frequencies of free ligand and their complexes [29, 30] helped with the assignments. A sharp band caused by the free azomethine υ-(C=N) ligand at 1648 cm^{-1} changes to a lower wave number and emerges at 1630–1636 cm^{-1}. It shows how coordination is affected by azomethine nitrogen. The phenolic group in the ligand, which causes a broad absorption band at 3403 cm^{-1}, was found to be missing in the mononuclear complexes. The absence of the phenolic group in metal complexes shows that phenolic hydrogen was deprotonated and that oxygen was coordinated with the metal. The phenolic C-O strength is seen in the free ligand as a relatively sharp band at 1453-1481 cm^{-1} [31]. Due to the coordination of oxygen with the metal ion, this frequency changes in the complex, moving towards the lower or higher values. It is possible to attribute bands at 668-694 cm^{-1} and 555-613 cm^{-1} to υ(M-O) and υ(M-N) vibrational frequencies that weren't present in Schiff bases [32]. The band at 3041–3060 cm^{-1} was assigned to the (-NH) group in ligands and it does not alter in metal complexes reveals that the (-NH) group did not participate in the complexation [33]. The results show that the bands in the ligand caused by (SO_2) remain same in the complexes indicating that this group is not coordinating with a metal center.

3.3. UV-Vis Spectroscopy

The various spectra of ligand and their metal complexes were measured in the concentration of 10^{-4} mol/L using a DMSO solvent at room temperature. The ligand shows mainly two absorption bands at 271 and 380 nm [32-35]. The band at 271 nm is due to $\pi \rightarrow \pi^*$ transition within the aromatic ring for ligand. The absorption band at 380 nm of ligand is assigned to $n \rightarrow \pi^*$ transition between lone pair electron of azomethine (-C=N-) group and a conjugated π bond of the aromatic unit. [36] The electronic transitions due to the organic ligand in the metal complexes, showed the absorption bands of the $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transition [37] results appeared at 262 nm and 284 nm regions for the azomethine (CH=N) and 330 and 334 nm regions for carbonyl (C=O) for copper and silver complex respectively. The absorption spectra of complexes are similar to each other which concern similarity in the structures of complexes. Upon complexation, these bands were shifted to lower wavelength, indicating the formation of metal complexes all the absorption value are listed in table 4.

Table 2. NMR Shifts of the ligand and complex.

Compound	Chemical Shift	(δ,ppm)	
$[L]H_2$	11.12(s,1H) 9.93(s.1H) 7.26(s,4H)	3.93(s,5H) 1.26(s,12H)	0.03(d, J=27.9 Hz ,4H)
[CuLX]	11.05(s,1H) 9.86(s.1H) 7.19(s,5H)	7.09(d, J=19.2Hz,3H) 6.91(s,5H) 3.86(s,5H)	1.51(s,29H) 1.19(s,65H) 0.78(s,26H) 0.00(s,27H)
[AgLX]	7.26(s,16H) 2.17(s,1H) 2.03(s,3H)	1.59(s,129H) 1.25(s,92H) 0.88(s,38H)	0.03(d, J=27.8 Hz ,30H)

Table 3. IR spectral data of Schiff base ligand $[L]H_2$ and its complexes (cm^{-1}).

Compounds	υ (OH)	υ(C=N)	υ (C-O)	υ (M-N)	υ (COO)	υ(M-O)
$[L]H_2$	3403	1648	1241	-	1357	-
[CuLX]	3078	1631	1256	541	1361	453
[AgLX]	3427	1630	1241	525	1387	604

Table 4. UV spectral data of ligand and complexes (nm).

Compounds	π → π*	n → π*
$[L]H_2$	271	380
[CuLX]	284	330
[AgLX]	262	364

Table 5. PXRD analysis of Cu(II) and Ag(II) complexes.

Compound	Formula	Temp.(K)	Wavelength	Radiation	Crystal system	2θ
Cu(II) Complex	$C_{11}H_{13}CuNO_{12}S_2$	298	1.540598	Cu Kα	Orthorhombic	10-90
Ag(II) Complex	$C_{22}H_{26}AgN_4O_{14}$	298	1.540598	Cu Kα	Rhombohedral	10-90

Table 6. Analysis of miller indices of Cu and Ag complex using JCPDS.

Complex	2θ	D (obs.)	h	k	l
Copper complex	18.729	4.734	0	1	1
	27.689	3.219	2	0	0
	46.432	1.9541	3	1	1
	49.022	1.8567	3	2	0
Silver complex	14.488	6.109	0	1	2
	20.956	4.236	1	1	0
	33.268	2.691	1	2	2
	45.252	2.002	3	1	2

3.4. Mass spectrometry

The fragmentation patterns with m/z have been identified from the TOF-MS ES+ spectral data of ligands and their complexes. [38]. The various spectra mass of the ligand and its copper and silver complexes were observed which shows its mass. The molecular ion peaks in the ligand spectra were located at m/z

224.4978 (calcd. 223.08) for Ligand due to [$C_{11}H_{13}NO_4$]. The mass spectra of complexes exhibited peaks at m/z value 476.87 (calcd. 478.89) for [$C_{11}H_{13}CuNO_{12}S_2$] and peaks at m/z value 679.5097 (calcd. 678.33) for [$C_{22}H_{26}AgN_4O_{14}$]. In the spectra of copper complexes the peak observed due to fragments $(C_{10}H_{13}CuNO_6S)^{+\cdot}$ at *m/z* 340.2582 (calcd. 338.82) and peak observed $(C_{10}H_{13}CuNO)^{+\cdot}$ at *m/z* 227.1745 (calcd. 226.77). In the spectra of silver complexes the peak observed due to fragments $(C_{19}H_{21}AgNO_5)^{+\cdot}$ at *m/z* 453.3427 (calcd. 451.25) and peak observed $(C_{17}H_{20}AgN_2O_2)^{+\cdot}$ at *m/z* 394.1040 (calcd. 392.23).

3.5. Powder X-ray diffraction studies

To identify the compounds structural characteristics, such as lattice parameters, unit cell volume and particle size, PXRD studies make it simple. The diffraction parameters of the copper and silver complexes have been confirmed and are depicted in table 5 and 6.

The X-ray diffraction of the complexes was plotted in the range 10-90°(2θ) at wavelength 1.540598 Å. The complexes clearly defined peaks in the XRD spectrum indicate that these metal complexes are crystalline. The Debye Scherrer formula is used to determine the complexes average crystalline size, as shown in equation 1 [38].

$$D = 0.91\ \lambda / \beta \cos \theta \text{----------Equation (1)}$$

where the value of constant 0.91 is the shape factor, k is X-ray wavelength of Cu Kα radiation (1.5406 Å), θ is represents Bragg diffraction angle and β is the full width at half maximum (FWHM).The above indexing method also gives Miller indices (h k l) unit cell parameters and unit cell volume. All the peaks were compared with the JCPDS. The value of these parameters of the respective two complexes are shown in Table 5.

4. Conclusion

The synthesis of copper (II) and silver (II) complexes using Schiff base is described in this article. Multiple spectral studies were used to characterize these complexes which were very helpful for understanding the structure and composition of complexes. The complexation of the metal ion with the ligand can be visualized using the UV and IR spectra. According to mass spectra the ligand and their complexes of copper (II) and silver (II), bind to each other in a 1:1 ratio and 1:2 ratio respectively. The amorphous nature of all synthesized complexes can be seen in the PXRD spectra of these complexes. The antibacterial, antifungal activity of these complexes were also examined and showed increase in effectiveness in complexes than the Schiff base ligand.

Authors Contribution

Siddharth Agrawal,Babita Agrawal Conceptualization, Methodology, Supervision, Manuscript Review, and Editing; Ranti Nur Aprillianti (RNA) Data Analysis, Manuscript Drafting; Vishnu Kumar Modanawal, Data Analysis; Arie Hardian (AH) Supervision, Manuscript Review and Editing. All authors approved and agreed for the submission of this article.

Conflicts of Interest

There is no conflict of interest between the authors.

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Abbreviations

o-vanillin; 2-Hydroxy-3-methoxybenzaldehyde

D (+) alanine; 2-((2-hydroxy-3methoxybenzylidene)amino) propanoic acid

[L]H2; Schiff Base Ligand

[CuLX]; opper (II)-Schiff Base Complex

[AgLX] ; Silver (II)-Schiff Base Complex

PXRD; Powered X-Ray Diffraction

PA; Pseudomonas aeruginosa

BC; Bacillus cirroflagellosus

PN; Penicillium notatium

AN; Aspergillus niger

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