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SYNTHESIS, CHARACTERIZATION AND ANTICANCER STUDIES OF GALLIUM (III) SCHIFF BASE METAL COMPLEXES

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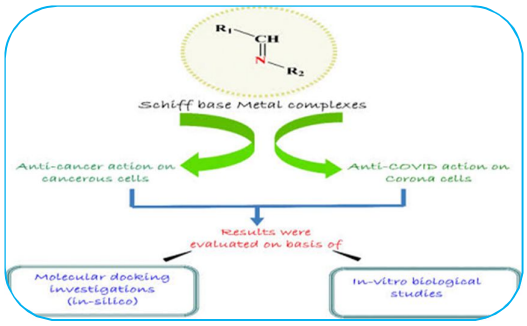
ABSTRACT

The Schiff base ligand 2- {[4- (4'- amino- phenoxy)- phenylimino]-methyl}-phenol [HL1], 2- {[4- (4'-amino- phenoxy)- phenylimino]-methyl}- naphthalene-2-ol [HL2], 2- {[4- (4'- amino- phenoxy)- phenylimino]-methyl}- 4 - bromophenol [HL3] and 2- {[4- (4'- amino- phenoxy)- phenylimino]-methyl}-6- methoxyphenol [HL4] and it's Ga (III) metal complexes had been synthesized. The ligand and metal complexes have been characterized with elemental analysis, UV-visible, IR, ^{1}H NMR spectroscopy, thermal studies, and magnetic susceptibility measurement. The crystallographic investigation reveals the tetragonal geometry of Ga (III) complexes of Schiff bases HL1 to HL4. Also, the magnetic moment and electronic spectral studies supported the tetrahedral structure Ga (III) complexes of Schiff bases. The antibacterial and antifungal activity of the Schiff base was accentuated while being coordinated with metal ions. Ligand [HL1-4] is immune to both human cancer cell lines, comprehensive of breast (MCF7) and colon (HT29) with GI50 of 64.2 M, 65.2 M, 75.2 M, and 88 M respectively. Ga (III) metal complexes confirm adequate activity in the breast (MCF7) cellular cell line with IC50 values of 33.1 M.



KEYWORDS: *Metal Complex, Thermal Properties, X-Ray Techniques, FTIR, Anticancer Activity.*

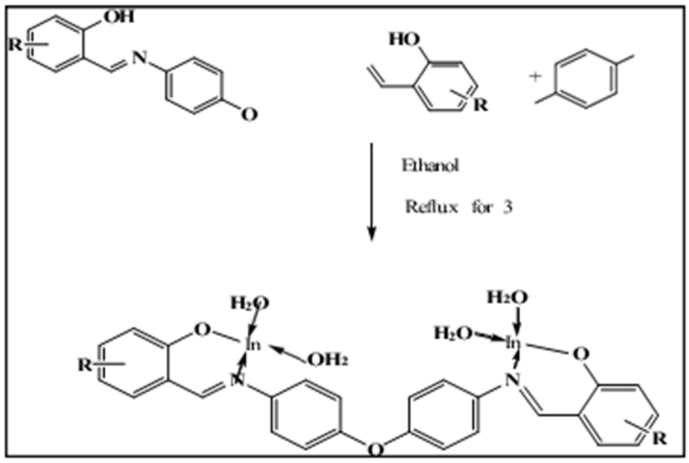
1. INTRODUCTION

The transition metal complexes with several donor sites were used in organometallic chemistry. A wide variety of Schiff base compounds were synthesized and structurally characterized.[1-4] The chemistry of transition metal complexes with multidentate[5-10] ligands has attracted interest because those metal ions can exhibit several oxidation states. The distinct oxidation states of these complexes confirmed a sturdy function in bioinorganic chemistry and redox enzyme systems. This may also provide the basis of models for energetic websites of biological systems or act as catalysts. Although several transition steel complexes[11-13] of Schiff bases have been structurally characterized notably few free Schiff bases have been similarly characterized. A combination of distinctly different steel ions binding with one ligand can lead to substances with exciting new properties[14-17] for instance, precise sensors, molecular wire, and magnetic and optical gadgets. The flexibility of Schiff base ligands can be improved by employing hydrogenation of their C=N bonds; they ought to as a result coordinate metallic ion circulate easily. For those reasons, the reduced Schiff base has recently received

giant interest. Schiff bases of 4-aminoantipyrine and its complexes are recognized for their variety of applications[18-23] within the areas of catalysis, medical uses, and pharmacologically. Antipyrine and its derivatives showed antibacterial and antitumor activities. Our studies became ongoing to establish the fact that nonbiologically active compounds come to be biologically active and much less biologically active become extra upon coordination/chelation with metallic ions. Metal complexes of monoanionic, both bidentate and pendant arm multidentate Schiff bases and related dianionic ligands inclusive of Salen (ethylenediamine bridged) and Salophen (o-phenylenediamine-bridged) have performed a completely crucial function inside the development of coordination chemistry for decades. In Schiff base complexes, the coordination surroundings at the metal center may be modified by attaching distinctive substituents to the ligand, which affords a useful style of steric and digital houses important for the exceptional tuning of structure and reactivity. Therefore, it's also not sudden that each transition-metallic and p-block metal derivative of Schiff bases was verified to catalyze a large shape of reactions.[24-26] Ga (III) complexes with a few salicylidene and β-hydroxy naphthylidene anilines. The formation of Ga (III) complexes with Schiff base occurs via bond formation between an indium ion and an unshared pair of electrons on azomethine nitrogen. While In (III) complexes with tripod Schiff base ligands through the condensation of substituted salicylaldehyde's with tris (2-aminoethyl) amine. Schiff bases supply several impartial complexes.[27-29] Our research become ongoing to set up the reality that nonbiologically compounds turn out to be biologically active, and less biologically lively end up extra upon coordination/chelation with metal ions. In the prevailing research, we synthesized the Schiff bases and their metal complexes which had been characterized by IR, NMR, molar conductance, magnetic moments, electronic elemental analysis, mass spectrum, and UV-visible spectroscopy. Schiff base ligands and their metallic complexes have been screened for their antimicrobacterial activity in opposition to Staphylococcus Aureus and the anticancer activity of the ligand (HL1-4) and its Ga (III) complexes changed into determined through sulforhodamine-B colorimetric assay.

2. EXPERIMENTAL

All reagents used had been pure AR grade such as 4,4'-Diaminodiphenylether, salicylaldehyde, 2-hydroxy-1-naphathaldehyde,5-bromo-2-hydroxybenzaldehyde,2-hydroxy-3-methoxy benzaldehyde, Indium trichloride, the solvents used have been ethanol, petroleum ether, ethyl acetate etc. The ligands have been prepared by previously published literature.[30] The 4,4'-diaminodiphenylether (2.00 g, 0.01mol) and salicylaldehyde (1.22 g, 0.01 mol) 2-hydroxy-1-naphathaldehyde (1.72g, 0.01mol), 5-bromo-2-hydroxy benzaldehyde, (2.00g, 0.01 mol), 2-hydroxy-3-methoxybenzaldehyde (1.52g, 0.01 mol), 2,4-dihydroxy benzaldehyde (1.38g, 0.01 mol) were dissolved in absolute ethanol (30-40 cm^3) separately in 1:2 molar ratio. The ethanolic solutions were mixed. After refluxing for 3 h, the solid crude product formed after cooling was recrystallized from ethanol and dried in desiccators over anhydrous $CaCl_2$. TLC was used to verify the purity of Schiff bases by using silica gel. A solution of 0.02 mole (4.4g) of gallium trichloride in ethanol (25 mL) blended with 0.01 mole (4.1g) of ligand HL_1 in DCM. The metal-to-ligand ratio had a molar ratio of 2:1. After 3 hours of refluxing, the solution formed a crude product after cooling. After purification, it was dried over anhydrous $CaCl_2$ [Scheme-1]. The same procedure is used for all Gallium complexes of Schiff bases, HL_2 (5.0g), HL_3 (4.7g), and HL_4 (4.6g).[31-32]



Scheme-1: Ga (III) Schiff Base Metal Complexes

3. RESULTS AND DISCUSSION

3.1 Elemental Analysis and Physical Properties

The elemental analysis of the Ga (III) Schiff base complexes is indexed in Table-1. The effects of carbon, hydrogen, and nitrogen percent are per the composition notified for the ligand and the complexes. The observed information simply agrees with the recommended molecular formula of all complexes. The statistics in addition advise that all these complexes are bis-binuclear and the metal-to-ligand ratio had a molar ratio of 2:1.

Table 1: Analytical and Physical Data of the Compounds Studied

Complex	Molecular formula	Molecular weight	C%	H%	N%	O%	In%
1	Ga2C26H26N2O7	707	44.10 (44.05)	3.65 (3.68)	3.94 (3.96)	15.83 (15.80)	32.45 (32.41)
2	Ga2C34H30N2O7	807	50.55 (50.56)	3.70 (3.66)	3.46 (3.48)	13.87 (13.90)	28.40 (28.38)
3	Ga2C26H24N2O7Br2	866	36.00 (35.98)	2.75 (2.72)	3.21 (3.25)	12.93 (12.91)	26.50 (26.51)
4	Ga2C28H30N2O9	767	43.80 (43.82)	3.90 (3.85)	3.63 (3.64)	18.77 (18.79)	29.90 (29.87)

3.2 Electronic Spectral Analysis

The strong absorption band determined under 300 nm is assigned to ligand π→π* transitions of the phenolic chromophores. The absorption band observed in all spectra within the range of 323-360 nm is most probably due to the transition of n→π* of the imine group.[33] Charge transfer bands are expected for Ga (III) complexes, which can be seen in the region of 370 - 465 nm.[34] The electronic absorption spectral information of the GaHL1 complex confirmed absorption bands at 230 nm and 246 nm, which can be assigned to π→π* transitions, and other at 354 nm which is assigned to the transition n→π* of the imine group. The electronic absorption spectral records of the GaHL2 complex showed absorption bands at 232 nm and 246 nm, which were assigned to π→π* transitions, and other at 336 nm the transition n→π* of the imine group. The electronic absorption spectral data of the GaHL3 complex showed three absorption bands at 236 nm and 248 nm, which can be assigned to π→π* transitions, and another at 360 nm which is assigned to the transition

n→π* of the imine group. The electronic absorption spectral data of the GaHL4 complex shows two absorption bands at 236 nm, which became assigned to π→π* transitions, and another at 360 nm which turned assigned to the n→π* transition of the imine group.

3.3 FTIR Spectral Analysis

The IR spectroscopic information represents feature vibrations bands of the ligands and metal complexes are shown in Table-2. The –OH becomes stretching and bending vibrational frequencies of the salicylaldehyde acting in the area 3500 – 3450 cm^{-1} and 1310 cm^{-1} respectively. The disappearance of these two peaks in the spectra of all the complexes indicates that the chelation takes place via the enolic – OH group. The OH stretching frequencies of the ligands are observed at about 3400 cm^{-1}. For the Ga (III) complexes, the disappearance of the band is anticipated due to the substitution of hydrogen for the metal inside the complicated formation. The strong bands at 1651 - 1642 cm^{-1} are characteristic of the azomethine (C=N) group present in the free ligand.[35] The ring skeletal vibrations (C=C) were in the region of 1503 - 1441 cm^{-1}.[36] On complexation, the bands were shifted to a lower frequency as compared to their free ligands indicating the coordination of atoms to the central metal ion.[37] In the complexes, weak bands in the 585 - 535 and 495 - 446 cm^{-1} range can be attributed to ν (M-N) and ν (M-O), respectively.[38] IR spectra of all Ga(III) complexes showed the presence of an extensive band at 3059 - 3172cm^{-1} which suggests that water is coordinated with the central metal ion.[39] Hence it can be concluded that each one of the complexes synthesized inside the present investigation incorporates an H_2O molecule in the coordination sphere. Ga (III) complexes do not show any absorption band at about 200 - 300 cm^{-1}. In IR spectra of Ga (III) complexes, the aromatic out-of-plane vibration is seen near 828 – 834 cm^{-1} and in-plane vibration at 740-750 cm^{-1}. The observation of new bands at 443 – 496 cm^{-1} and 580 –525 cm^{-1} region are due to ν(M–O) and ν(M–N)[40] is in addition proof of coordination.

Table-2: IR Spectral Data of Indium Metal Complexes.

Complex	*ν*(OH)	*υ* (C=N)	υ (M N)	*υ* (M O)	*ν*(Ar)
$Ga_2C_{26}H_{26}N_2O_7$	3114	1620	544	443	2860
$Ga_2C_{34}H_{30}N_2O_7$	3059	1621	530	490	2872
$Ga_2C_{26}H_{24}N_2O_7Br_2$	3172	1617	525	470	2850
$Ga_2C_{28}H_{30}N_2O_9$	3115	1613	580	496	2835

3.4 Thermogravimetric Analysis

The thermogram of the Ga (III) complexes of ligands (HL1, HL3, HL4) confirmed four decomposition steps within the temperature range of 100 - 985°C. The first step of decomposition, within the temperature range of 40 - 225°C, corresponds to the loss of four water molecules. In the subsequent steps, 1st, 2nd, 3rd decomposition occurs within the range of 200 – 400°C, 400 - 575°C, and 575 - 985°C respectively which correspond to the removal of the organic part of the ligands step by step, leaving metal oxide as a residue. The thermogram of the Ga HL_2 complexes showed five decomposition steps within the temperature range of 40 - 985 °C (Fig.-1). The metal percentages calculated from metal oxide or metal residues were compared with those determined by the analytical metal content determination.[41]

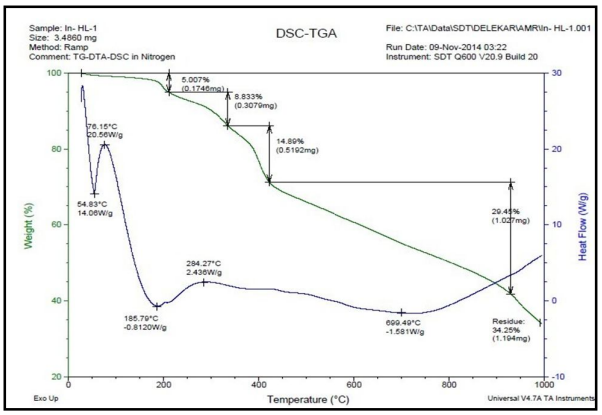


Fig.-1: Thermal Analysis of Gallium Complex

3.5 X-ray Diffraction Analysis

The X-ray diffraction patterns indicate that the ligand and its complexes are crystalline, with various degrees of crystallinity as shown in Fig.-2. Some of the extra peaks present in the complexes compared to ligands prove the coordination of metal ions.[42] The residue obtained after the thermal treatment of metal complexes was subject to X-ray diffraction analysis and patterns obtained. It was found that the diffraction patterns obtained match the corresponding metal oxide diffraction patterns. The detailed X-ray diffraction studies of the selected complexes. Based on these observations, the structure of all Ga (III) complexes of Schiff bases, HL_1 to HL_4 is confirmed to be tetragonal structures. From all these studies, it is clear that the ligand acts as a bidentate towards metal ions.

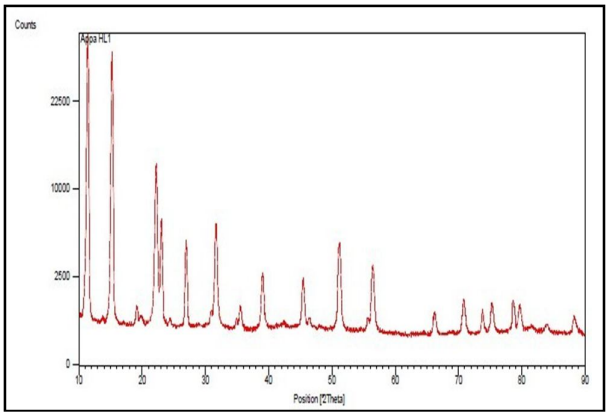


Fig.-2: X-Ray Diffraction Pattern of $GaHL_1$ Metal Complex

3.6 Anticancer Activity

The anticancer activity of the ligand (HL1-4) and its Ga (III) complexes were determined by sulforhodamine-B colorimetric assay. RPMI 1640 medium was used to culture the cell lines, along with 10% fetal bovine serum (FBS) and 2 mM L-glutamine, at 37°C in a humidified atmosphere with 5% CO_2. A culture medium was used to seed about 5×10^{3} cells per well in a 96-well microtiter plate. After 24 hours, Schiff base (HL1-4) and its Ga (III) metal complexes at the concentration of 10, 20, 40, and 80 µg mL^{-1}Molar

were added to respective wells and incubated for 48 hrs. After incubations, the sulforhodamine-B assay was performed.[43] Ligand [HL1-4] is able to resist both human cancer cell lines such as breast (MCF7) and colon (HT29) with a GI50 of 62.2 M, 65.2 M, 72.2 M, and 84 M respectively. Moderate activity is present in Ga (III) metal complexes for the breast (MCF7) cell line with an IC50 value of 31.1 M.

4. CONCLUSION

The molar conductance value exhibits the presence of electrolytic compound and does not use an inorganic anion outside the coordination sphere. The magnetic moment and electronic spectral research suggest a tetrahedral structure. The XRD statistics also show the tetragonal geometry of Ga (III) complexes of Schiff bases HL1 to HL4. The spectral data demonstrated that oxygen and nitrogen atoms are involved in the complexation process. From the TGA-DSC, it was clear that each complex incorporates four water molecules. Because of the above facts, a tetrahedral structure is proposed for gallium (III) complexes of Schiff bases HL1 to HL4. Ligand [HL1-4] is able to resist both human cancer cell lines such as the breast (MCF7) and colon (HT29) with GI50 of 62.2 M, 65.2 M, 72.2 M and 84 M respectively. The breast (MCF7) cell line exhibits moderate activity in Ga (III) metal complexes with an IC50 value of 34.1 M.

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