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Green Synthesis, Characterization and Biological Investigation of Cr (II), Cu (II), and Fe (III) Complexes with Schiff Base Ligand Derived from Substituted Salicylaldehyde

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ABSTRACT

A new pyrimidine derivative Schiff base ligand has been synthesized by the condensation of 2-amino-4-hydroxy-6-methylpyrimidine and 3-bromo-5-chlorosalicylaldehyde. Metal complexes of the Schiff base were prepared by the reaction of the Schiff base and Iron nitrate in ethanol solution. The complexes isolated, washed and dried. The Schiff base is pale yellow, while Cr(II), Cu(II), And Fe(III) complexes is light yellow. The synthesized compounds have been characterized by FT-IR, 1H-NMR and UV-Vis techniques for the ligands and FT-IR, UV-Vis, all reactions monitored by TLC, molar conductivity and magnetic susceptibility measurements for the corresponding complexes.The complexes is paramagnetic. The results of the molar conductivity measurements indicated that all complexes are non-electrolytes in (DMSO). An octahedral geometry for all the complexes of. The ligands are bidentate, (L) through phenolic (OH) and azomethine nitrogen. The ligand and its complexes were screened for their antifungal and antibacterial activity against Aspergillus Niger, Penicillium chrysogenum, Fusarium Moniliform, Aspergllus Flavus and Escherichia Coli, Salmonella Typhi, Staphylococcus Aureus, B. subtilis. The result indicated that the complexes exhibited good antifungal and antibacterial activities.

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Introduction

The pyrimidine nucleus is an integral part of biomolecules like DNA and RNA and plays an important role in several biological processes and also has considerable pharmacological uses such as antibiotics, antibacterial, cardiovascular as well as agrochemical and veterinary product [1]. By considering all this importance we are synthesized fused pyrimido pyrimidine and its derivatives with simple path.

The Heterocyclic compounds are plenteous in nature and are of great significance to life because their structural subunits exist in many natural products such as vitamins, hormones, and antibiotics hence, they have to drug considerable attention in the synthesis of biologically active molecules and advanced organic chemistry [2-4]. Also, in the group of heterocyclic compounds nitrogen containing heterocycles are an important class of compounds in the medicinal chemistry and also contributed to the society from biological and industrial applications [5]. A totally unsaturated membered six-ring containing nitrogen is known as azine or pyridine; with two nitrogen atoms it is known as diazine, and with a nitrogen at 1,2-position, it is known as pyridine, at 1,3 positions as pyrimidine and at 1,4 positionas pyrazine [6]. However, the current review intends to study and deep investigation on the significance of pyrimidine class of antimicrobial agents along with clinical and in vitro applications of pyrimidine derivatives to accessibility the development of more potent as well as effective antimicrobial agents [7].

Pyrimidine is the heterocyclic aromatic compound similar to benzene, pyridine containing nitrogen atom at position 1 & 3 of six membered ring. Heterocyclic ring pyrimidine moiety are of great interest because thy constitute an impotence class of natural and non-natural product, which many of exhibit useful biological activity and clinical application [8]. Substituted Purines, pyrimidine occur very widely living organisms. Pyrimidines reside in an important position in the medicinal chemistry as it has a number of diverse biological properties. Their related fused heterocycles like pyramid pyrimidine and its derivatives were of interest as potential bioactive molecules. Pyrimidine derivatives are reported to have diverse pharmacological activities such as anticonvulsant, antihypertensive activity, analgesic, anti-depressive, antipyretic, anti-inflammatory, Chemotherapeutic agents, antiviral, anti-HIV, antimicrobial and anti-tumour activities [9-18].

In presence of pyrimidine base thiamine, cytosine and uracil which are the essential therapeutic application. The literature survey indicated that a wide range of pharmacological activities are exhibited by the compound encompassing pyrimidine nucleus. In addition to this, various analogy of pyrimidine has been found to possess antibacterial, antifungal, antioxidant, antihistaminic,

antiallergy agents, antiviral, anticancer activities and also act as calcium channel blockers [19-24].

A search of literature revels that no work has been done on the transition metal complexes of the Schiff bases derived from 2-amino-4-hydroxy-6-methylpyrimidine and 3-bromo-5-chlorosalicylaldehyde. In this communication we report the synthesis of bidentate Schiff bases formed by the condensation of 2-amino-4-hydroxy-6-methylpyrimidine and 3-bromo-5-chlorosalicylaldehyde (Figure 5). The solid complexes of Co(II), Fe(III), and Mn(II) with these ligands have been prepared and characterized by different physico-chemical methods.

Materials and Methods

Reagents and Solvents

The 2-amino-4-hydroxy-6-methylpyrimidine (Aldrich sigma), 3-bromo-5-chlorosalicylaldehyde, metal nitrate of AR grade was used for synthesis of ligand and metal complex.

Synthesis of Ligand

The ligand was prepared by a modification of the reported methods [25]. The Schiff base ligand has been synthesized by refluxing a mixture of 0.01 mol (1.2015g) of 3-bromo-5-chlorosalicylaldehyde and 0.01 mol (1.2710 g) of 2-amino-4-hydroxy-6-methylpyrimidine in 50 ml super dry ethanol refluxed for about 4h. Schiff base thus formed was cooled to room temperature and collected by filtration, followed by recrystallization in ethanol and dried in vacuo over anhydrous calcium chloride (Yield:71%).

Synthesis of Metal Complexes

To a hot ethanol solution (25ml) of the ligand (2 mol) and (25ml) of metal Nitrate (1mol) was added with constant stirring. The pH of reaction mixture was adjusted to 7-8 by adding 10% alcoholic ammonia solution and refluxed for about 3 h. The precipitated solid metal complex was filtered off in hot condition and washed with hot ethanol and dried over calcium chloride in vacuum desiccators (Yield: Cr=62%, Cu=67%, Fe=75%).

Physical Measurement

IR spectra were recorded on FTIR(ATR)-BRUKER -TENSOR37 spectrometer using KBr pellets in the range of 4000-400 cm^{-1}. 1HNMR Varian mercury 300MHZ spectra of ligand were measured in CDCl3 using TMS as internal standard. X-RD were recorded on BRUKER D8 Advance. TGA- DTA were recorded on Shimadzu. The carbon, hydrogen and nitrogen contents were determined on Elementar model vario EL-III. The UV-visible spectra of the complexes were recorded on model Jasco V-530 UV-Vis spectrometer. Molar conductance of complexes was measured on Elico CM 180 conductivity meter using 10^{-4} M solution in DMSO. Magnetic susceptibility measurements of the metal chelates were done on a Guoy balance at room temperature using $Hg[Co(SCN)_4]$ as a calibrant.

Results and Discussion

Schiff bases of 2-amino-4-hydroxy-6-methylpyrimidine and its complexes have a variety of applications including biological, clinical and analytical. The coordinating possibility of 2-amino-4-hydroxy-6-methylpyrimidine has been improved by condensing with a variety of carbonyl compounds. An attempt has been made to synthesize Schiff bases from 2-amino-4-hydroxy-6-methylpyrimidine with 3-bromo-5-chlorosalicylaldehyde. Physical characteristics, micro analytical, and molar conductance data of ligand and metal complexes are given in (Table 1 and 2). The analytical data of complexes revels 2:1 molar ratio (ligand: metal) and corresponds well with the general formula $[ML_2(H_2O)_2]$ [where M= Cr(II), Cu(II), And Fe(III)] . The magnetic susceptibilities of Cr(II), Cu(II), And Fe(III) complexes at room temperature are consistent with high spin octahedral structure with two water molecules coordinated to metal ion. The presence of two coordinated water molecules was confirmed by TGA-DTA analysis. The metal chelates solutions in DMSO show low conductance and supports their non-electrolyte nature (Table 1).

Molar Conductivity Measurements

The metal (II) complexes were dissolved in DMSO and the molar conductivity of 10-4 M of their solution at room temperature was measured. The lower conductance values of the complexes support their non-electrolytic nature of the compounds.

Table 1: Physical Characterization, Analytical and Molar Conductance Data of Compounds

Compound Molecular formula	Mol.Wt.	M.P. Decomp temp. 0C	Colour	Molar Conduc. Mho. Cm^2mol^{-1}
L	265	95	Yellow	---
Cr-L	589	>300	Dark Yellow	11.20
Cu-L	598	>300	Yellow	13.22
Fe-L	607	>300	Yellow	11.26

Table 2: Elemental Analysis of Co(II), Fe(III), and Mn(II) Complex

Compound	% Found (Calculated)			
	C	H	N	M
L	63.54 (63.23)	3.33 (3.32)	15.60 (15.85)	----
Cr-L	51.50 (51.53)	3.48 (3.40)	13.17 (13.16)	9.81 (9.87)
Cu-L	52.46 (52.52)	3.54 (3.52)	16.17 (16.16)	10.71 (10.73)
Fe-L	53.43 (53.50)	3.34 (3.37)	17.10 (17.12)	10.91 (10.93)

^{1}H-NMR Spectra of Ligand

The ^{1}H-NMR. Spectra of free ligand at room temperature show the following signals. 2.35 δ (s, 3H, Methyl hydrogen bonded to pyrimidine ring), 2.35 δ (s, 3H, Methyl hydrogen bonded to phenyl ring), 5.47 δ (s, 1H, Phenolic (OH) hydrogen of pyrimidine ring), 6.77 δ(s, 1H, Hydrogen bonded to pyrimidine ring), 7.84 δ (s, 1H, hydrogen bonded to azomethine carbon), 7.2-7.42 δ (D,4H, Aromatic Ha, Hb, protons of phenyl ring).

IR Spectra

The IR spectra of the complexes are compared with that of the ligand to determine the changes that might have taken place during the complexation.The bands at 3363, 1678, 1516, 1309, and 1186 cm-1assignable to OH (intramolecular hydrogen bonded), C=C(aromatic), C=N (azomethine), C-N (aryl azomethine) and C-O (phenolic) stretching modes respectively [26-28]. The absence of a weak broad band in the 3200-3400 cm^{-1}region, in the spectra of the metal complexes suggests deprotonation of the intramolecular hydrogen bonded OH group on complexation and subsequent coordination of phenolic oxygen to the metal ion. This is further supported by downward shift in υ C-O (phenolic) with respect to

free ligand. On complexation, the (C=N) band is shifted to lower wave number with respect to free ligand, denoting that the nitrogen of azomethine group is coordinated to the metal ion. The C-N band is shifted to lower wave number with respect to free ligand, The IR spectra of metal chelate showed new bands in between the 500-600 and 400-500 cm^{-1}regions which can be assigned to M-O and M-N vibrations respectively The IR spectra of CO (II) show a strong band in the 3050-3600 cm-1region, suggesting the presence of coordinated water in these metal complexes. The presence of coordinated water is further confirmed by the appearance of non-ligand band in 830-840 cm-1region, assignable to the rocking mode of water [29-31]. The presence of coordinated water is also established and supported by TGA/DTA analysis of these complexes. Hence it is concluded that the coordination takes place via phenolic oxygen and azomethine nitrogen of ligand molecule.

Thermogravimetric Analysis

The dynamic TGA with the percentage mass loss at different steps have been recorded. The simultaneous TGA/DTA analysis of Cr (II) was studied from ambient temperature to 1000^0C in nitrogen atmosphere using α-Al_2O_3 as reference. An analysis of the thermogram of the complexes indicated that Cr (II) complexes ligandL (Figure 1) show two step decomposition. The first weight loss 5.61 % , in between temp. 50-1950C could be correlated with the loss of two molecules of lattice water (calcd 6.50 %). The anhydrous compound does not remain stable at higher temperature, it undergoes rapid decomposition in the range 195-570°C, with 79.45 % mass loss corresponds to decomposition of the complex (calcd. 79.14 %) in second step. The decomposition is completed leading to the formation of stable residue of metal oxide CrO obs. 11.23 % (calcd. 14.35 %). kinetic and thermodynamic viz the energy of activation (Ea), frequency factor (Z), entropy change (-ΔS) and free energy change (ΔG) for the non-isothermal decomposition of complexes have been determined by employing Horowitz-Metzger method values are given in Table 3 [32]. The Calculated values of the given activation energy of the complexes are relatively low, indicating the autocatalysis effect of metal ion on the thermal decomposition of the complex. The negative value of activation entropy indicates that the activated complexes were more ordered than the reaction was slow. The more ordered nature may be due to the polarization of bonds in the activated state, which might occur through charge transfer transitions [33].

Table 3: The Kinetic and Thermodynamic Parameters for Decomposition of Metal Complexes

Complex	Step	Decomp. Temp. (^{0}C)	n	Ea (kJmole^{-1})	Z (S^{-1})	ΔS (JK-1mole^{-1})	ΔG (kJmole^{-1})	Correl -ation coefficient
Cr-L	I	369	1.3	11.88	2.26 x 10^4	-167.97	24.86	0.933
Cu-L	I	373	1.4	11.78	2.23 x 10^4	-168.95	24.76	0.923
Fe-L	I	381	1.2	11.74	2.25 x10^4	-169.15	24.79	0.928

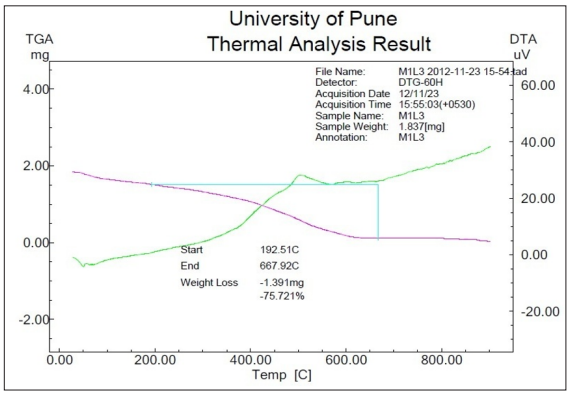


Figure 1: TGA-DTA Curve of Cu(II) Complex of Ligand L

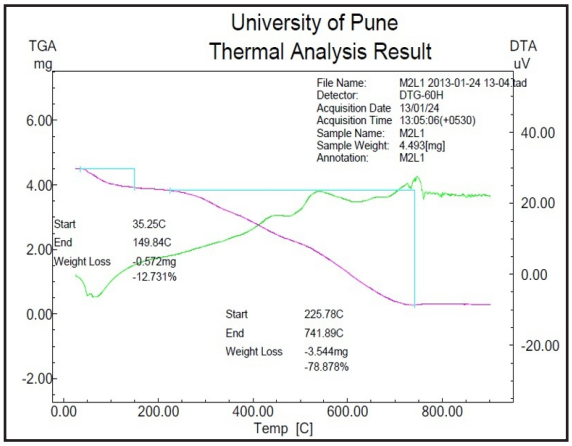
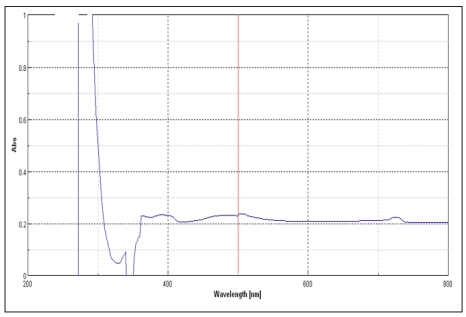


Figure 2: TGA-DTA Curve of Cr(II) Complex of Ligand L

Magnetic Measurements and Electronic Absorption Spectra

The electronic spectral studies as shown below Figure 3 of metal complexes of Fe (III) with Schiff bases were carried out in DMSO solution. The absorption spectrum of the Fe(III)complex shows bands at 13812 $cm^{-1}$and 30030 cm^{-1} are assigned to $^2B_{1g} \rightarrow 2A_{1g}$ and charge transfer respectively in an octahedral field [34]. The Ni (II) complexes were diamagnetic in nature.



Wavelength	Absorption
778	0.205
742	0.206
724	0.226
502	0.239
484	0.233
402	0.233
392	0.235
362	0.230
340	0.092

Figure 3: Electronic Absorption Spectra of Fe(III) Complex of Ligand L

Powder X-Ray Diffraction

The x-ray diffractogram of Cr (II) complexes of L8 was scanned in the range 20-80° at wavelength 1.543 Å (Figure 4). The diffractogram and associated data depict the 2θ value for each peak, relative intensity and inter-planar spacing (d-values). The diffractogram of Mn(II) complex of L had fifteen reflections with maxima at 2θ = 12.89° corresponding to d value 6.86Å. The x-ray diffraction pattern of these complexes with respect to major peaks of relative intensity greater than 10% has been indexed by using computer programmed the above indexing method also yields Miller indices (hkl), unit cell parameters and unit cell volume [35]. The unit cell of Cr(II) complex of L yielded values of lattice constants, a= 9.76 Å, b=10.31 Å, c = 27.02 Å and unit cell volume V=2718.90393 Å3. In concurrence with these cell parameters, the condition such as a = b = c and α = β = γ = 900required for sample to be Monoclinic were tested and found to be satisfactory. Hence it can be concluded that Cr(II) complex has Orthorhombic crystal system. Hence it can be concluded Cr (II) complex of L has monoclinic crystal system. The experimental density values of the complexes were determined by using specific gravity method and found to be 1.0664 $gcm^{-3}$for Cr (II) complexes respectively [31]. By using experimental density values, molecular weight of complexes, Avogadro's number and volume of the unit cell were calculated. Number of molecules per unit cell were calculated by using equation ρ = nM/NV and was found Cr (II) complexes respectively. With these values, theoretical density was computed and found to be 1.0554 $gcm^{-3}$for respective complexes. Comparison of experimental and theoretical density shows good agreement within the limits of experimental error [33].

Table 4: Indexed X-Ray Diffraction Data of Cr(II) Complex of Ligand L

Peak No.	2θ (observed)	2θ (calculated)	d (observed)	d (calculated)	Miller indices of Planes			Relative intensities (%)
					h	k	l	
1	6.56777	6.54182	6.74738	6.76126	0	0	4	70.32
2	7.8503	7.82903	5.64859	5.65492	0	1	4	46.54
3	9.09671	9.07831	4.8788	4.88198	2	0	0	12.91
4	11.19744	11.20684	3.97107	3.96343	2	1	3	18.99
5	13.43783	13.44986	3.3177	3.31177	2	0	6	100
6	14.49187	14.46597	3.0808	3.0836	3	1	1	28.10
7	16.38805	16.36481	2.73221	2.73396	0	3	6	33.33
8	17.74172	17.74805	2.52955	2.52696	1	1	10	12.21
9	19.9865	19.98566	2.25501	2.25375	0	0	12	20.25
10	22.59581	22.57324	2.00584	2.0067	1	4	8	1.90
11	24.47211	24.47079	1.86039	1.8596	2	5	3	4.51
12	27.92459	27.88619	1.64553	1.64693	5	3	4	2.59
13	29.27174	29.24678	1.57602	1.57663	4	2	12	2.88
14	30.61782	30.59087	1.51299	1.51364	4	2	13	5.70
15	31.79153	31.78755	1.46265	1.4623	6	3	2	2.02
16	34.22879	34.23567	1.36986	1.36918	6	4	2	1.57

Unit Cell Data and Crystal Lattice Parameter

a (A^0) = 9.76 Volume(V) = 2718.90393 $(A^0)^3$
b (A^0) = 10.31 Density(obs.) = 1.0664 gcm^{-3}
c (A^0) = 27.02 Density(cal.) = 1.0554 gcm^{-3}
α = 90.00 Z = 3
β = 90.00 Crystal system = Orthorhombic
γ = 90.00 Standard deviation (%) = 0.072 Porosity = 1.04%

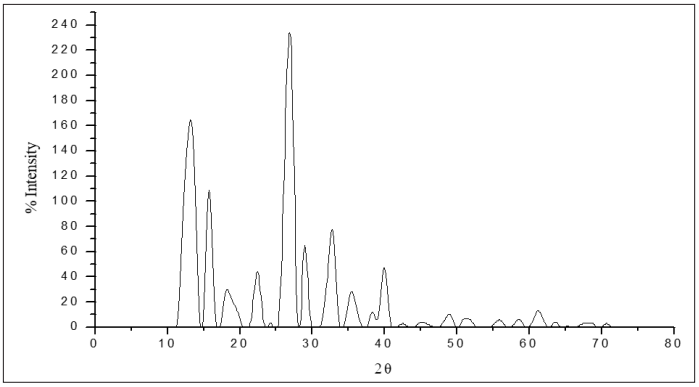


Figure 4: X-ray Diffractogram of Cr (II) Complex of L

Antibacterial Activity

Antifungal activity and Antibacterial activity of ligand and metal complexes were tested in vitro against fungal such as Aspergillus Niger, Penicillium chrysogenum, Fusarium moneliforme, Aspergillus flavus and bacteria such as E. Coli, B.Subtilis, S. Aurious And Bacillus subtlis by paper disc plate method The compounds were tested at the concentrations 1% and 2% in DMSO and compared with known antibiotics viz Griseofulvin and Penicilin [36-39]. (Table 6 and 7)., it is found that the inhibition by metal chelates is higher than that of a ligand and results are in good agreement with previous findings with respect to comparative activity of free ligand and its complexes [40].

Table 6: Antifungal Activity of Ligands

Test Compound	Antifungal Growth							
	Aspergillus niger		Penicillium chrysogenum		Fusarium moneliforme		Aspergillus flavus	
	1%	2%	1%	2%	1%	2%	1%	2%
L	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
Cr-L	-ve	-ve	-ve	RG	-ve	-ve	-ve	+ve
Cu-L	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
Fe-L	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
+ve control	+ve	+ve	+ve	+ve	+ve	+ve	+ve	+ve
-ve control (Griseofulvin)	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve

Ligand& Metal : +ve – Growth (Antifungal Activity absent)
-ve - Growth (Antifungal Activity present)
RG - Reduced Growth (More than 50% reduction in growth observed)

Table 7: Antibacterial Activity of Ligands and their Metal Complexes

Test Compound	Diameter of inhibition zone (mm)							
	E. coli		Salmonella typhi		Staphylococcu saureus		Bacillus subtlis	
	1%	2%	1%	2%	1%	2%	1%	2%
L	19mm	23mm	18mm	24mm	17mm	20mm	19mm	24mm
Cr-L	17mm	25mm	16mm	24mm	19mm	24mm	22mm	26mm
Cu-L	18mm	23mm	19mm	22mm	21mm	25mm	17mm	24mm
Fe-L	15mm	20mm	20mm	26mm	22mm	27mm	18mm	25mm
DMSO	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
Penicillin	19mm	19mm	23mm	23mm	27mm	27mm	26mm	26mm

Ligand & Metal: - ve - No Antibacterial Activity
Zone of inhibition - --mm

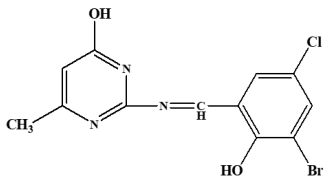


Figure 5: Structure of Schiff Base Ligand L

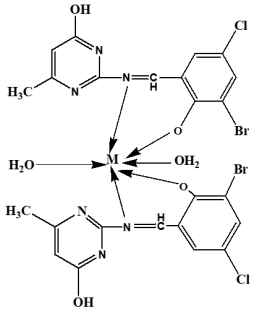


Figure 6: The proposed Structure of the Metal Complexes. [When M= Cr(II), Cu(II), and Fe(III)]

Conclusion

In the light of above discussion, we have proposed octahedral geometry for Cr(II), Cu(II), and Fe(III) complexes. On the basis of the physico-chemical and spectral data discussed above, one can assume that the ligand behave as dibasic, NO bidentate, coordinating via phenolic oxygen and imino nitrogen as illustrated in Fig.6. The complexes are biologically active and show enhanced antimicrobial activities compared to free ligand. Thermal study reveals thermal stability of complexes. The X-ray study suggests orthorhombic crystal system for Co(II), Fe(III), and Mn(II) complexes.

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