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Synthesis and Antimicrobial Evaluation of Schiff Bases Derived from 2-amino-4, 6-dimethyl benzothiazole with Pyrole-2-aldehyde, Pyridine-2-aldehyde and their Metal Complexes

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Abstract: ***The studies of 12 transition metal complexes of Schiff bases are discussed, Schiff bases are obtained by condensation of 2-amino-4,6-dimethyl benzothiazole with Pyrrole 2-aldehyde, Pyridine-2-carbaldehyde and Cu(II), Co(II), Ni(II), Fe(II), Mn(II) and Cr(II) chloride were chosen to synthesize new complexes. The Schiff bases and the complexes were characterized on the basis of physicochemical studies viz. spectral studies like IR, ^{1}H NMR, Mass studies. The ligands and their metal complexes were screened for antibacterial activity against various bacteria like Escherishia Coli, Salmonella typhi, Staphylococcus aureus, Bacillus subtilis and fungicidal activity against various fungi like Aspergillus niger, Penicillium chrysogenum, Fusarium moneliforme, Aspergillus flavus.***

Keywords: Benzothiazole, Schiff bases, metal complexes, spectral study, antimicrobial activity

I. INTRODUCTION

Schiff bases are known to possess variety of biological properties. They are usefull synthones for many compounds. They can be used for synthesis of bioactive molecules. Schiff bases derived from an amino and carbonyl compounds are an important class of ligands that coordinate to metal ions via azomethine nitrogen and have been studied extensively. Schiff bases of hydroxyl aldehydes and ketones were widely used in co-ordination chemistry for the preparation of metal complexes. In azomethine derivatives the C=N linkage is essential for biological activity, several azomethine were reported to possess remarkable antibacterial, antifungal, anticancer and diuretic activites. Some of them have been used as complexing agent and powerful corrosion inhibitors. A Schiff base of hydroxy acetophenone and its complexes has a variety of applications in biological, clinical, analytical and pharmacological areas. Earlier work has shown that some drugs showed increased activity when administered as metal chelates rather than as organic compounds and that the co-ordinating possibility of hydroxyl acetophenone has been improved by condensing with a variety of carbonyl compounds

Here in this paper we report the synthesis of Schiff bases as ligand and their metal complexes Cu(II), Co(II), Ni(II), Fe(II), Mn(II) and Cr(II) . All the synthesized compounds were screened for their antimicrobial activity. Further the structures of synthesized compounds were confirmed by elemental analysis and spectral studies.

II. EXPERIMENTAL

All the melting points were determined in an open capillary tube and are uncorrected; completion of the reaction was monitored by thin layer chromatography on precoated sheets of silica gel G. All the reagents used were chemically pure and are of AR grade. The ligand selected in the preparation of metal complexes are (4,6-Dimethyl-benzothiazol- 2-yl)-(1H-pyrrol-2-ylmethylene)-amine(L_1),(4,6-Dimethyl-benzothiazol-2-yl)-pyridine-2-ylmethylene-amine(L_2). Transition metals Cu(II), Co(II), Mn(II), Fe(II), Cr(II) and Ni(II) were used for the synthesis of metal complexes with corresponding Schiff base ligands.

A. Synthesis of Schiff base (4,6-Dimethyl- benzothiazol-2-yl)-(1H-pyrrol-2-ylmethylene)-amine(L_1)

Pyrole-2-aldehyde and 2-amino, 4, 6-dimethyl benzothiazole, were dissolved in distilled ethanol in equimolar quantities. The reaction mixture was refluxed on water bath for about 4-5 hours: the progress of the reaction was monitored by TLC. The hot reaction mixture was then poured on ice cold water the solid thus separated was filtered washed with water and dried. The solid then recrystallized from ethanol.

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B. Synthesis of Schiff base (4,6-Dimethyl- benzothiazol-2-yl)-pyridine-2-ylmethylene-amine (L_2)

Pyridine-2-aldehydeand 2-amino, 4, 6-dimethyl benzothiazole, were dissolved in distilled ethanol in equimolar quantities. The reaction mixture was refluxed on water bath for about 4-5 hours: the progress of the reaction was monitored by TLC. The hot reaction mixture was then poured on ice cold water the solid thus separated was filtered washed with water and dried. The solid then recrystallized from ethanol.

C. Preparation of Complexes:

The Metal salt (0.01 moles) and the ligand (0.02 moles) in 50ml ethanol were mixed. The P^H of the mixture solution was raised up to 5 using alcoholic ammonia. The solution was then concentrated on steam bath in a china bowl. Solid complex thereafter separate out washed with acetone to remove excess of ligand and dried over $CaCl_2$.

III. RESULT AND DISCUSSION

All the complexes at room temperature are insoluble in water and the most of the common organic solvent but soluble in DMF and DMSO. The analytical data of the complexes (Table-1) indicate that their stoichiometry may be represented as 1:2 metal to ligandratio.

Table 1: Physical data of Synthesized Ligands and Metal complexes

Entry	Molecular Formula	Color	Yield (%)	Melting Point (°C)
L1	C14H13N3S	Faint Yellow	86	115°C
L1M1	[Cu(C14H12SN3)2].nH2O	Hena	64	190°C
L1M2	[Co(C14H12SN3)2].nH2O	Vannilla	74	>280°C
L1M3	[Ni(C14H12SN3)2].nH2O	Blue	68	>280°C
L1M4	[Fe(C14H12SN3)2].nH2O	Brown	66	>280°C
L1M5	[Mn(C14H12SN3)2].nH2O	Grey	67	262°C
L1M6	[Cr(C14H12SN3)2].nH2O	Grey	62	>280°C
L2	C15H13N3S	Brown	78	110°C
L2M1	[Cu(C15H13SN3)2].nH2O	Parrot Green	64	274°C
L2M2	[Co(C15H13SN3)2].nH2O	Green	65	272°C
L2M3	[Ni(C15H13SN3)2].nH2O	Brown	62	254°C
L2M4	[Fe(C15H13SN3)2].nH2O	Grey	68	182°C
L2M5	[Mn(C15H13SN3)2].nH2O	Dull Yellow	71	>280°C
L2M6	[Cr(C15H13SN3)2].nH2O	Green	66	>280°C

L1 and L2 are ligands M1 = Cu, M2 =Co, M3 =Ni, M4 =Fe, M5 = Mn, M6=Cr

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A. Spectroscopic Data of Selected Compounds

1) Schiff base (L_1)

IR (KBr): 1612cm^{-1}(C=N), 3186 cm^{-1} 2916 cm^{-1}(CH_3) , ^{1}HNMR: δ 2.3 (s,3H), δ 2.5(s,3H), δ 6.3 (s,1H), δ 7.0-7.7 (m,5H);M.S. (m/z): 255

2) Schiff base (L_2)

IR(KBr): 1585cm^{-1}(C=N),2913cm^{-1}(N-H), 3340 cm^{-1}

(CH_3)^{1}HNMR: δ2.0(s,3H),δ2.5(s,3H), δ 5.2 (s,1H), δ 6.4-7.0 (Ar-H) ; M.S. (m/z):267

The IR spectra of all the synthesized complexes were compared with those of the free ligand in order to determine the coordination sites that may be involved in coordination. Upon comparison it was determined that the *ν*(C=N) stretching vibration is found in the Schiff base at 1612 in L_1 and 1585 in L_2 cm^{-1}. This band shifted to lower wave numbers in the complexes indicating the participation of nitrogen in coordination. The new bands at *ν* M-N stretching vibrations were appeared at 560- 525 cm^{-1} in the spectra of metal complexes.^{1}H NMR spectra of the transition complexes were recorded, unfortunately, however due to the presence of a metal ion, proton resonance was not affected and one could observe only broad peaks indicating the formation of the complex.

IV. ANTIMICROBIAL ACTIVITY

A. Antibacterial Activity Procedure

The antibacterial activity was measured by agar cup method. Nutrient agar (Himedia) was prepared and sterilized at 15 Psi for 15 minutes in the autoclave. It was allowed to cool below 45 °C and seeded with turbid suspension of test bacteria separately, prepared from 24 hours old slant cultures. 3% inocula were used every time. The bacterial cultures selected were, two gram negative cultures viz. Escherichia coli, salmonella typhi and two Gram positive cultures Viz;Staphylococcus aureus, Bacillus subtilis. This seeded preparation was then poured in sterile petri plate under aseptic condition and allowed it to solidify Cups of 10 mm diameter were borered in the agar plate with sterile cork borrer. 100 µl of compound solution prepared in the cup under aspectic condition with the help of micropipette. 100µl of DMSO was also placed in one of the cup as blank (negative control). A standard antibiotic disk impregnated with 10 units of penicillin was also placed on the seeded nutrient agar surface as standard reference antibiotic (positive control).The plates were kept in refrigerator for 15 minutes to allow diffusion of the compound from agar cup into the medium. Then the plates were shifted to incubator at 37 °C and incubated for 24 hours.

After incubation plates were observed for the zone of inhibition of bacterial growth around the agar cup. Results were recorded by measuring the zone of inhibition in millimeter (mm) using zone reader. Antibacterial Activity of the synthesied ligand and their metal complexes are given in table.

Table 2: Antibacterial activity of the synthesized ligand and their metal complexes

S.No.	Compound	Escherishia coli	Salmonella typhi	Staphylococcus aureus	Bacillus subtilis
1	L1	-Ve	-Ve	-Ve	-Ve
2	L1M1	22 mm	21 mm	25 mm	26 mm
3	L1M2	21 mm	20 mm	34 mm	30 mm
4	L1M3	19 mm	18 mm	21 mm	26 mm
5	L1M4	19 mm	22 mm	23 mm	20 mm

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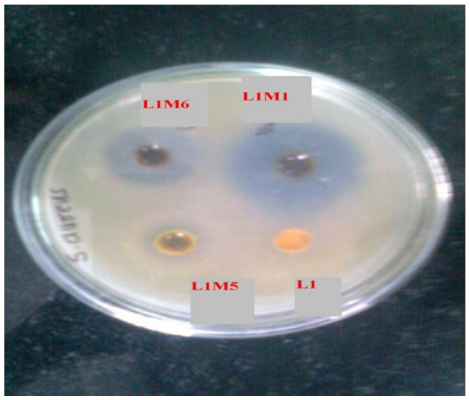
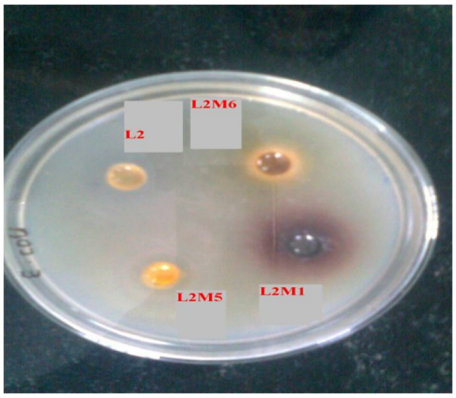
6	L1M5	18mm	13	25 mm	22 mm
7	L1M6	13	17 mm	31mm	30 mm
8	L2	-Ve	19 mm	22mm	22 mm
9	L2M1	28 mm	24 mm	31 mm	37 mm
10	L2M2	18 mm	20 mm	28 mm	39 mm
11	L2M3	-Ve	23 mm	28 mm	37 mm
12	L2M4	22 mm	23 mm	27 mm	23 mm
13	L2M5	18 mm	17 mm	31 mm	30 mm
14	L2M6	18 mm	16 mm	18 mm	15 mm
	DMSO	-ve	-ve	-ve	-ve
	Peniciline	13 mm	18 mm	36 mm	18 mm

Ligands - -ve = No antibacterial activity Zone of inhibition in mm





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B. Antifungal Activity Procedure

Antifungal activity was measures by poison plate method. The medium which was used is Potato Dextrose Agar (Himedia). The medium is prepared and sterilized at 10 Psi in autoclave for 15 minutes. Then the compound which is to be tested is added to the sterile medium in aspectic condition so as to get the final concentration as 1%. A plate with DMSO was prepared as blank (negative control). Similarly a plate with 1% Gresiofulvin was prepared as standard reference plate (positive control).

To test fungal cultures Aspergilus niger, Penicillium Chrysogenum, Fusarium moneliforme, Aspergillus flavus these were selected. They were allowed to grow on slant for 48 hours so as to get profuse speculation. 5ml of 1:100 aqueous solution of tween 80 was added to the slant and spores were scraped with the help of nicrome wire loop to form suspension.

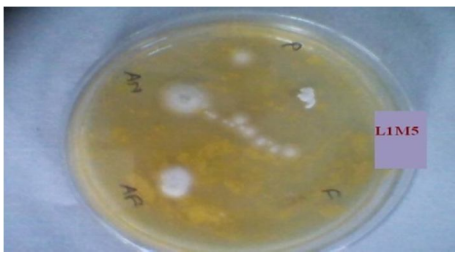
With the help of nicrome wire loop the fungal suspension was spot inoculated on the plate's prepared using compound. The plates were incubated at room temperature for 48 hours. After incubation plates were observed for the growth of inoculated fungi. Results were recorded as growth of fungi (no antifungal activity) reduced growth of fungi(moderate antifungal activity) and no growth of inoculated fungi (antifungal activity).

Antifungal activity of all the synthesized ligand and their metal complexes are given in the following table.

Table 3: Antifungal activity of the synthesized ligand and their metal complexes

S. No.	Compound	Aspergillus niger	Penicillium chrysogenum	Fusarium moneliforme	Aspergillus flavus
1	L_1	+Ve	+Ve	+Ve	+Ve
2	L1M1	-Ve	-Ve	-Ve	-Ve
3	L1M2	-Ve	-Ve	-Ve	-Ve
4	L1M3	-Ve	-Ve	-Ve	-Ve
5	L1M4	+Ve	-Ve	-Ve	+Ve
6	L1M5	-Ve	-Ve	-Ve	-Ve
7	L1M6	-Ve	-Ve	-Ve	-Ve
8	L2	-Ve	-Ve	-Ve	-Ve
9	L2M1	-Ve	-Ve	-Ve	-Ve
10	L2M2	-Ve	-Ve	-Ve	+Ve
11	L2M3	-Ve	-Ve	-Ve	-Ve
12	L2M4	-Ve	-Ve	-Ve	-Ve
13	L2M5	-Ve	-Ve	-Ve	-Ve
14	L2M6	-Ve	-Ve	-Ve	-Ve
	+Ve control	+Ve	+Ve	+Ve	+Ve
	-Ve control (Griseofulvin)	-Ve	-Ve	-Ve	-Ve

Ligands +Ve - Growth (No AntifungalActivity)
-Ve – No Growth (Antifungal Activity Observed)



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V. CONCLUSION

From the present result it is clear that preliminary studies showed their good inhibitory properties. In general the transition metal complexes of the corresponding Schiff bases are more active than their parent ligand and hence may serve as vehical for activation of the ligand as principle cytotoxic species.

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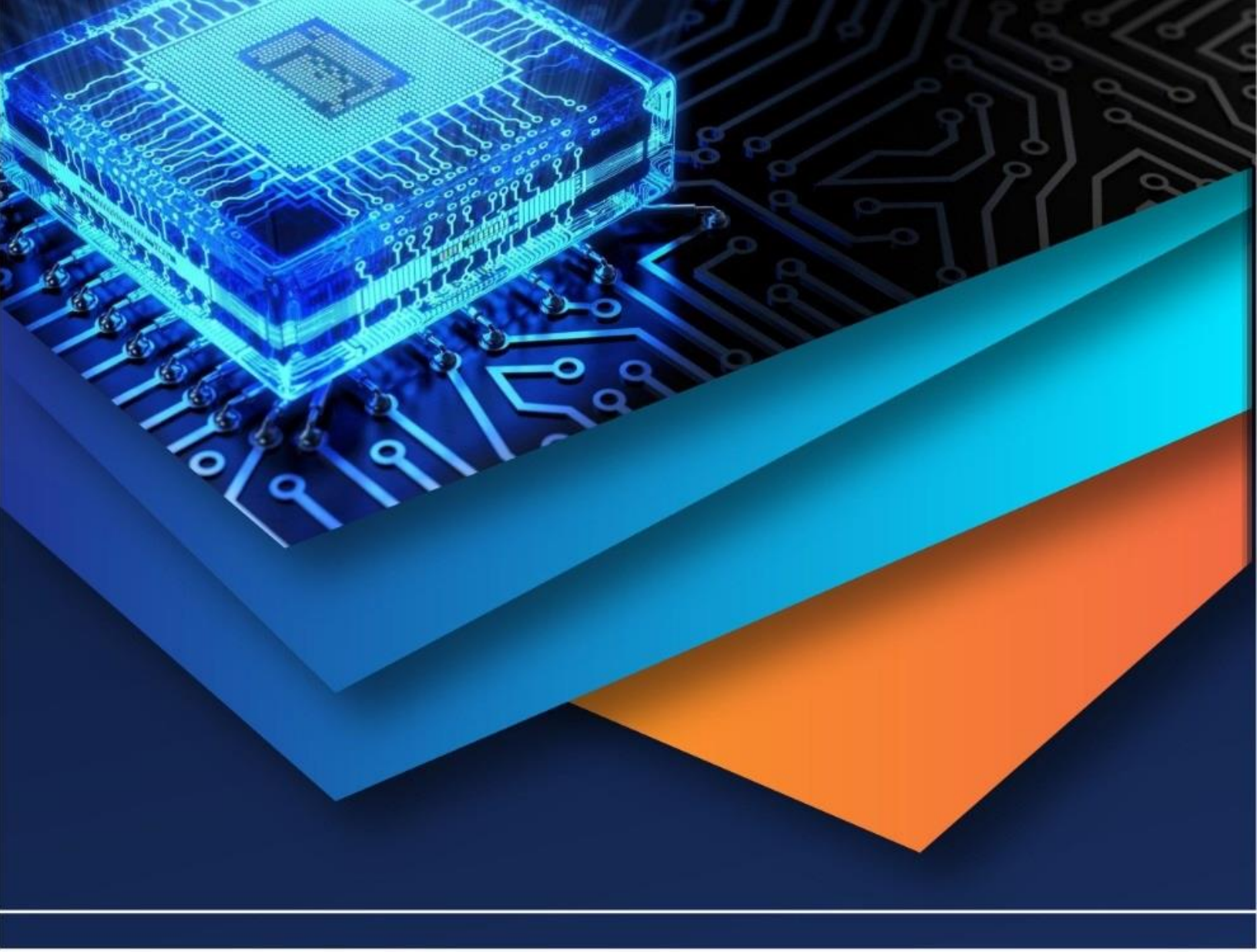

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