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Synthesis, Evaluation of Structure of Antimicrobial Sulpha Drugs by Spectroscopic Techniques

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ABSTRACT
Some new Cu (II), Zn (II), Mn (II) and Cr (III) complexes of Schiff base derived from Sulpha drugs have been synthesized. They have been characterized on the basis of elemental analysis, IR and ^{1}HNMR spectroscopy. The spectral data suggests that Schiff base acts as bidentate ligand and an octahedral environment exist around the metal ion. They are potential antimicrobial agents against a goup of microbes as follows, Esherichia coli, Salmonella typhi, Bacillus subtilis, Staphylococcus aureus, Aspergillus flavous, Aspergillus niger, Penicillium citrinum and Fusarium oxysporum.
Keywords: Cu (II), Zn (II), Mn (II) and Cr (III) complexes, IR and ^{1}HNMR spectroscopy, antimicrobial activity and Analytical Instrumentation.

1. INTRODUCTION

Sulpha drugs, were the first antibacterial antibiotics , and paved the way for the antibiotic revolution in medicine. Sulpha drugs are a group of compound which show distinct physical, chemical and biological properties.[1] Many chemotherapentically important sulpha drugs like sulphadiazine, sulphathiazole, sulphamerazine and sulphonamide possess SO_2NH_2 moiety which has an important toxicophoric function.[2] The Schiff bases synthesised from the sulpha drug possess pronounced antimicrobial[7], antitumour[3], antifouling[10], antioncogenesis activities[4]. The condensation products of sulpha drugs with aldehydes and ketones are biologically active and also have good complexing ability; their activity increases an complexation with metal ions[5].
The enhanced activity of metal complexes of Schiff bases derived from sulpha drugs has led to considerable interest in their coordination chemistry. A survey of literature reveals that very little attention has been paid on coordination behaviour of Schiff bases derived from sulpha drugs. The newly prepared complexes of sulpha drugs were characterized on the basis of elemental analysis and spectral studies. These complexes were also screened for their fungicidal and bactericidal activities.

2. EXPERIMENTAL & INSTRUMENTATION

All the chemicals used were of AR grade. The liquid reagents were purified by distillation.
p'- Hydroxybenzylidene-p-aminobenzene sulphonamide (SD) was synthesized[6] by refluxing on water bath Sulphonamide (17.2g, 0.1 mole) and aldehyde (12.2g, 0.1 mole) in 75 ml alcohol for two hours. On cooling the Schiff base (SA) separated out as white shining crystals. It was filtered, washed, dried and finally recrystallised from hot ethanol solution.
The Cu (II), Zn (II), Mn (II) and Cr (III) complexes of Schiff base were prepared[7] by refluxing on water bath, a mixture of 0.01 mole of metal chloride (i.e. 1.70 g of $CuCl_2.2H_2O$, 1.36 g of $ZnCl_2$, 1.97 g of $MnCl_2.4H_2O$ and 2.66 g of $CrCl_3.6H_2O$) in 15 ml ethanol respectively with 0.02 mole of Schiff base SA (i.e. 5.52 g of SD) in 20 ml ethanol for 3 hours. The reaction mixture was concentrated and then cooled. The solid derivatives were separated out. These were filtered, washed and finally air-dried.
For the microanalysis of C, H and N, CHN Perkin-Elmer micro analyzer 240 was used. The metal contents of the complexes were analyzed by standard methods. The IR spectra of the Schiff base and its metal complexes were recorded on Perkin-Elmer 4250 spectrophotometer in the range 4000-200 cm^{-1} in CSI/KBr matrix. The ^{1}HNMR spectra of the Schiff base and the metal complexes were recorded in $CDCl_3$ on a Bruker DRX 300F, 300 MHz FTNMR spectrometer.

Antibacterial Activity

The bactericidal activity was evaluated by the filter paper disc diffusion method[8]. The nutrient agar medium (peptone, beef extract, NaCl and agar-agar) and 5mm diameter paper disc of Whatman No.1 were used. The compounds were dissolved in DMF in 100, 250 and 500 ppm concentrations. The filter paper discs were soaked

in different solutions of the petridishes already seeded with the test organisms. The plates were incubated for 24 hours at 37ºC and the inhibition zone around each disc was measured.

Antifungal Activity

The fungicidal activity was evaluated by Agar plate techinique[9,10]. The fungi were grown in agar medium (glucose 20g, starch 20g, agar-agar 20g and 1000ml water) at 28 ± 2ºC and the compounds after being dissolved in DMF were mixed in the medium. The growth of the fungus was obtained by measuring the diameter of colony in petridishes after 3 days and the percentage inhibition was calculated by the formula.

$$\% \text{ inhibition} = \frac{(C-T)\times 100}{C}$$

Where C and T are the diameters of the fungus colony in control and test plate, respectively.

3. RESULTS AND DISCUSSION

The obtained metal complexes of the Schiff base are coloured solid, insoluble in water but soluble in benzene, acetone, DMF and other polar organic solvents. Elemental analyses suggests 1:2 (M:L) Stoichiometry[20]. All the physical and analytical results are listed in Table-1.

Table-1.Physical & Analytical Data of Schiff base (SD) and its Metal Complexes

COMPOUND	MOLECULAR WEIGHT	COLOUR	M.P. (ºC)	ELEMENTAL ANALYSIS % FOUND (CALCULATED)				
				C	H	N	S	METAL
SD $[C_{13}H_{12}O_3N_2S]$	276	Off-White	170-172º	56.40 (56.52)	4.30 (4.34)	10.02 (10.14)	11.25 (11.59)	- -
Cu-SD [Cu $(C_{26}H_{24}N_4O_6S_2)$. $2H_2O$]	651	Golden Brown	144º	47.77 (47.92)	3.60 (3.68)	8.44 (8.60)	9.62 (9.83)	9.77 (9.92)
Zn-SD [Zn $(C_{26}H_{24}N_4O_6S_2)$. $2H_2O$]	653	Cream	157º	47.63 (47.77)	3.63 (3.67)	8.44 (8.57)	9.77 (9.80)	9.54 (9.67)
Mn-SD [Mn $(C_{26}H_{24}N_4O_6S_2)$. $2H_2O$]	643	Light Brown	175º	4 8.44 (48.52)	3.34 (3.73)	8.71 (8.70)	9.74 (9.80)	9.88 (9.95)
Cr-SD [Cr $(C_{26}H_{24}N_4O_4S_2)$. $2H_2O$]	640	Light Green	166º	48.45 (48.75)	3.57 (3.75)	8.41 (8.75)	10.04 (10.00)	8.17 (8.12)

Infra Red Spectra

The Infra red spectra of the Schiff base and the metal complexes show sharp bands in the region 3250-3300 cm^{-1} and 1575-1590 cm^{-1} attributed to the stretching and bending vibrations respectively of $\nu(NH_2)$. While the symmetric and asymmetric vibrations of $\nu(SO_2)$ are observed in the region 1150-1210 cm^{-1} and 1310-1335 cm^{-1} respectively. The IR spectra of Schiff base show a sharp band at 1621cm^{-1} attributed to ν(C=N) azomethine linkage , a shift of ± 15 cm^{-1} is observed in this frequency in case of metal complexes which is suggestive of the coordination of the metal ion with the azomethine linkage. The band in the far IR spectra of metal complexes in the region 530-560 cm^{-1} are tentatively assigned to ν(M-N) vibrations. The stretching and bending vibrations found in the region 660-695cm^{-1} and 830-860 cm^{-1} respectively are attributed to the coordination of water molecules. The IR-spectral data is tabulated in Table-2.

Table-2.Tentative assignments of some selected bands in the IR spectra (cm^{-1}) of Schiff base (SD) and its metal complexes.

Compounds	v (NH_2) (strech.)	v(NH_2) (bend.)	v(SO_2) (sym.)	v(SO_2) (asym)	v (C=N)	v(H_2O) (stretch)	v(H_2O) (bend.)	v (M-N)
SD	3275	1561	1168	1348	1629	-	-	-
Cu-SD	3171	1568	1160	1328	1655	679	830	552
Zn-SD	3286	1577	1152	1318	1620	657	842	555
Mn-SD	3297	1583	1155	1339	1639	653	835	549
Cr-SD	3301	1585	1172	1326	1647	621	824	555

^{1}HNMR Spectra

The ^{1}HNMR spectra of the Schiff base and its metal complexes show multiplet signals in the region 6.65-8.40 ppm due to benzene ring protons. While the signals due to HC=N protons appear at 4.76 ppm in the spectra of Schiff base while it shifts downfield in the spectra of corresponding metal complexes. The broad signals due to $–NH_2$ protons also shift downfield in the spectra of metal complexes. Thus, this downfield shift is attributed to the complex formation i.e. attachment of metal with nitrogen atom of $–NH_2$ group and nitrogen atom of HC=N group. The evidences mentioned above supports the complex formation of the Schiff base. The ^{1}HNMR data is tabulated in Table-3.

Table-3.^{1}HNMR Spectral data of Schiff base (SD) and its metal complexes

S.No.	Compound	Chemical Shift, S.(ppm)	Peak position	Group Assigned.
1.	SD	(a) 6.57-8.43 (b) 4.19 (c) 10.16	Multiplet Singlet Singlet	Aromatic ring HC=N $-NH_2$
2.	Cu-SD	(a) 6.84 -8.22 (b) 4.06 (c) 10.27	Multiplet Singlet Singlet	Aromatic ring HC=N $-NH_2$
3.	Zn-SD	(a) 6.27-7.48 (b) 4.54 (c) 10.44	Multiplet Singlet Singlet	Aromatic ring HC=N $-NH_2$
4.	Mn-SD	(a) 6.47-8.41 (b) 4.09 (c) 10.26	Multiplet Singlet Singlet	Aromatic ring HC=N $-NH_2$
5.	Cr-SD	(a) 6.26-8.24 (b) 4.72 (c) 10.11	Multiplet Singlet Singlet	Aromatic ring HC=N $-NH_2$

On the basis of spectroscopic characterization the tentative structure of Schiff base (SD) and its metal complexes is given in Figure-1

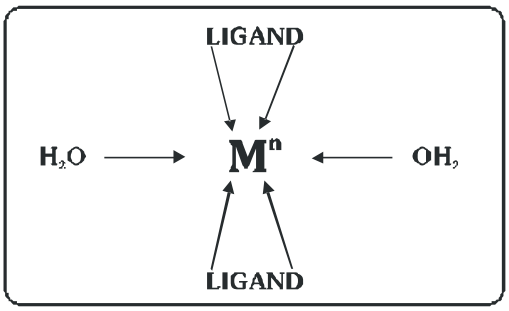

Fig 1. Tentative structure of the metal complexes

where M^n = Cu (II), Zn (II), Mn (II) and Cr (III) metal ions

LIGAND = Schiff Base SD

Antibacterial and Antifungal Activities

The Schiff base (SA) as well as the metal complexes exhibited good amount of activity against all the tested bacteria and fungi. The Schiff base derived from Sulpha drugs are moderately active against all the tested bacteria and fungi. In comparison to the Schiff base, their corresponding metal complexes cause more inhibition, i.e. more activity. The Schiff base (SD) along with its metal complexes showed maximum activity against the

bacteria, *E. coli* and minimum against the bacteria, *S. aureus*. Though, the fungicidal studies reveal that the activity was maximum against *A. niger* followed by *F. oxysporum, A flavous* and *P. citrinum*.
In general the activity of the metal complexes was in following order :-

Cr (III) > Mn (II) > Zn (II) > Cu (II).

4. CONCLUSION

The increased activity of metal complexes may be due to the effect of the metal ion configuration and the charge on normal cell. A possible mode of toxicity may be specified by Chelation Theory. Chelation considerably reduces the polarity of the metal ion mainly because of partial sharing of its -electrons and delocalization over the whole chelate ring. Such chelation increases the lipophilic character of metal chelate, which probably tends to break down the permeability barriers of cells, resulting in the interference with the normal cell process. Thus, the results suggest that the metal complexes of Schiff base (SD) have proved to be excellent bactericides and fungicides in the present investigations.

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