

Synthesis, Physical Characterization and Antibacterial Evaluation of M (III) Schiff Base Complex



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Abstract

The M (III) Schiff base complexes synthesized from Schiff base ligand prepared by the condensation of salicylaldehyde and o-amino benzoic acid. Metal selected for the preparation of complexes was Cr(III), Fe (III) and Mn (III). Hence, there are four metal complexes were synthesized. The chemical structure of the synthesized metal ligand complexes were confirmed IR and NMR spectral analysis. The free Schiff base and its complex have been tested for their antibacterial activity against several pathogenic bacteria, such as *Pseudomonas aeruginosa*, *Proteus vulgaris*, *Proteus mirabilis*, *Klebsiella pneumonia* and *Staphylococcus aureus*. The antibacterial activity was determined by the Agar Ditch technique using DMF (polar) and 1, 4 dioxane (non polar) as solvent.

Keywords: Schiff Base, Antibacterial Activity, DMF, 1,4 Dioxane, Salicylaldehyde, O-aminobenzaldehyde.

Introduction

Schiff bases are involved as intermediates in the processes of non-enzymatic glycosylations. These processes are normal during aging but they are remarkably accelerated in pathogenesis caused by stress, excess of metal ions or diseases such as diabetes, Alzheimer's disease, and atherosclerosis. Non-enzymatic glycosylation begins with an attack of sugar carbonyls or lipid peroxidation fragments on amino groups of proteins, amino phospholipids and nucleic acid, causing tissue damages by numerous oxidative rearrangements. One of the consequences is cataract of lens proteins [1]. Many biologically important Schiff bases have been reported in the literature possessing, antimicrobial, antibacterial, antifungal, anti-inflammatory, anticonvulsant, antitumor and anti HIV activities [2-7]. Transaminases are found in mitochondria and cytosol of eukaryotic cells catalyzed by a class of enzymes. Most of the work in the field of coordination chemistry describes mainly four, five, six or seven coordinate compounds of transition and inner transition metals ions mono, bi, tri, tetra and multidentate open chain organic ligands. Compound which on dissolution do not give ion of which they are made but instead give complex ion are called co-ordination compounds. Co-ordination compounds exhibit different characteristic properties which depend on the metal ion to which they are bound. The nature of the metal as well as the type of ligand etc. these metal complexes have found extensive application in various fields of human interest. Cr(III), Mn(III), and Fe(III) complexes as well as ligand were tested for their antibacterial and antifungal properties against some pathogen (*Escherichia coli*, *Staphylococcus aureus*, *Aspergillus niger* and *Fusarium oxysporum*). The antibacterial activity of the Schiff base and its Cr(III) was tested on gram positive and gram negative bacteria, the magnetic moment value of the prepared complex revealed the existence of a diamagnetic character [8-10]

Aim of the Study

In the present work, complexes of Cr (III), Fe (III), and Mn (III) with Schiff base have been synthesized, characterized the chemical structure by IR and NMR spectral analysis and to study the antibacterial activity of the prepared Schiff base complex derived from salicylaldehyde and o-amino benzoic acid.

Review of Literature

The nature of the co-ordination depends on the metal ion and the donor atoms, as well as on the structure of the ligand and the metal ligand interaction. O-amino benzoic acid and salicylaldehyde compounds are capable to form complex with transition metal ion in the form of Schiff base the complex of Cr (III), Mn (III) and Fe(III) with two Schiff base have been synthesized. Their antibacterial activity towards some clinically important bacteria was evaluated [11-13]. Some Schiff base complexes derived from salicylaldehyde and histidine with some divalent transition metal ion. In the prepared complexes Cd(II) Schiff base complex showed greater antibacterial activity [14]. Cr (III), Mn (III) and Fe(III) metal complexes of new heterocyclic Schiff base derived from 1-amino-5-benzoyl-4-phenyl-1 H-pyrimidine-2-one with salicylaldehyde have been prepared and investigated by elemental analysis, mass, electronic, IR and ¹H NMR spectra. Octahedral geometry was suggested for all complexes [15, 16]. Cr (III), Mn (III) and Fe(III) complexes of Schiff base derived from istain with some amino acids were synthesized and identified on the bases of their chemical analysis using IR and electronic spectra. All the complexes were suggested to possess an octahedral geometry [17]. A novel Schiff base ligand derived from 2,2 bis (P-methoxyphenylamine), salicylaldehyde and its transition metal complexes with Cr (III), Mn (III) and Fe(III) ion prepared and their spectral properties were investigated [18]. The complex of Cr (III), Mn (III) and Fe(III) ion with a Schiff

and primary amines have been prepared and investigated using different chemical techniques, such as elemental analysis, electronic spectra. The obtained chemical analysis data showed the formation of 1:1 (metal:ligand) ratio and the square planar geometry was suggested for Cr (III), Mn (III) and Fe(III) complexes and an octahedral for Cr(III) and Fe(III) complexes [19].

M(II) complexes of Schiff base have been tested against some pathogenic microorganism, a comparative study of the ligands and complexes indicate that the complexes exhibit higher antibacterial activity than the free ligand and control [20].

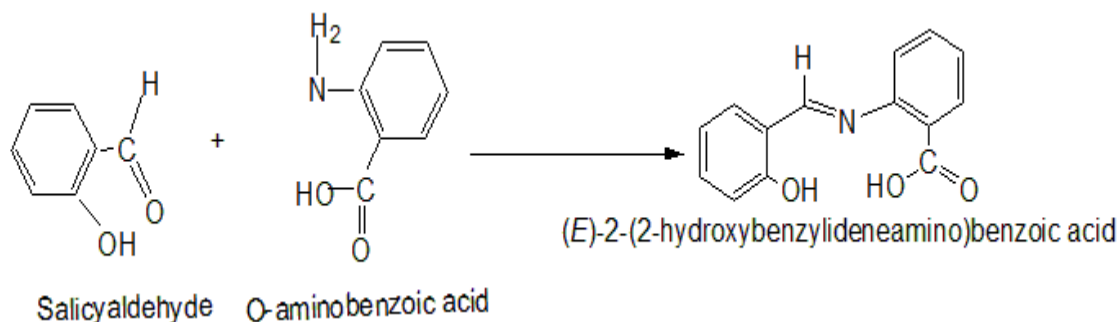
Material and Method

Metal salts of trivalent metal, Dimethyl formamide (DMF) and 1,4- dioxane were purchased from Sud. Chem. India. Salicylaldehyde and O-aminobenzoic acid were purchased from Fluka. The Antibacterial activity of synthesized Schiff base metal complexes was determined by Agar-ditch method.

Experimental

Preparation of Schiff Base

20 ml ethanolic solution of salicylaldehyde (1.22g: 0.01mol) and the same volume of ethanolic solution of O-amino benzoic acid (1.37g: 0.01mol) are mixed. The mixture was stirred for 3-4 hour. This solution was evaporated under vacuum to remove the solvent. The product after filtration washed several time with ethanol and recrystallized from hot ethanol and dried under vacuum the colour of the product is orange and its purity was confirmed by chromatography technique.



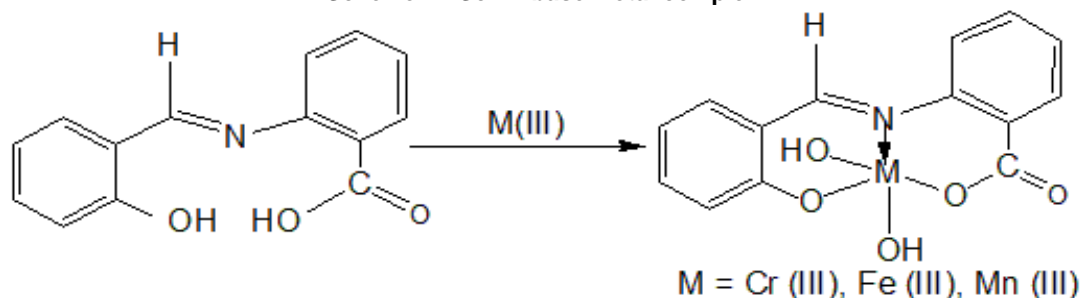
base derived from 1,4-dimethylamino benzaldehyde

Scheme I: Synthesis of Schiff Base Ligand Synthesis of the complex

For each metal complex, different metal salt solution were prepared. The compounds used for the synthesis of the Mn, Cr, and Fe complexes were cupric chloride, nickel chloride, ferrous ammonium sulphate and zinc chloride, respectively. A mixture of the Schiff base under investigation (0.01mol, 2.41g) 20cm³ ethanol and the same amount of the same solvent of

M(III) salt (0.01mol, 2.37g) was refluxed for 4-5 hours in a water bath the PH of the solution was maintained by the buffer solution. A colour precipitate was obtained. The precipitate was filtered and washed several time with hot ethanol to remove excess metal ion, respectively. The precipitate was then dried and stored in a desiccator over anhydrous CaCl₂ under vacuum.

Scheme II: Schiff base metal complex



Result and Discussion

¹H NMR

A survey of literature reveals that Schiff base have characterized by ¹H NMR and ¹³C NMR spectra to ensure ligand structure and purity in d₆-dimethylsulfoxide (DMSO-d₆) solution using Me₄Si (TMS) as internal standard. The ¹H NMR spectra of the ligand shows broad signal at 9.4-12.1 ppm due to the presence of -NH [21] and 2.1-2.8 ppm due to the -CH₂- (cyclic) [22]. The multiples in the region 6.54-8.76 ppm may be assigned to aromatic proton [23, 24]. ¹³C NMR of the Schiff base ligand, the signal appeared in the region 113-158 are assigned to aromatic carbon. The signal at 182.8-171.2 and 165.4-150.7 due to C=N and C=O respectively.

Electronic Spectral Studies, Magnetic Measurements and Molar Conductance

The electronic spectra of Cr (III) complexes showed absorption band in the region 8970-9310, and

27530-27820 cm⁻¹ attributed to 4B_{1g}→4E_{1g}, 4B_{1g}→4B_{2g}, 4B_{1g}→4A_{2g} and 4B_{1g}→4E_g. The spectral bands are consistent with that of five coordinated Cr (III) complexes [25, 26]. The magnetic moment values for these complexes were found to be 3.58-4.81 B.M. [27].

The absorption spectral bands of manganese (III) complexes showed three spin allowed transitions: 5B_{1g}→5A_{1g}, 5B_{1g}→5B_{2g}, 5B_{1g}→5E_g appearing in the ranges 12235-12640, and 35360-35520 cm⁻¹. The magnetic moment values for these complexes were found in the range 4.81-5.62 B.M [27].

The electronic spectra of the iron (III) complexes gave two bands at 9940-9990, and 27440-27650 cm⁻¹, which could be assigned to the transitions 6A_{1g}→4T_{1g} and 6A_{1g}→4T_{2g}, respectively, suggesting a five coordinated square pyramidal geometry of Fe (III) complexes [28]. The complexes show magnetic moment values in the range 5.10-5.40 B.M [29].

Table 1 The structure of the metal complexes was confirmed by I.R (KBr, Cm⁻¹) analysis

Complex	-OH (Stretching)	-OH (Bending)	-C=N (Stretching)	M-N	M-O
Cr(III) Schiff base Complex	3341	1350	1511	744	530
Fe(III) Schiff base Complex	3306	1354	1539	750	949
Mn(III) Schiff base Complex	3136	1332	1565	685	475

Table 2 Analytical data (CHN) of divalent Cr, Fe and Mn Schiff Base Complexes

Complex	C	H	N	M	Colour	Magnetic moment
Cr(III) Schiff base Complex	51.70 (51.68)	3.41 (3.38)	4.31 (4.29)	15.99 (15.95)	Orange	3.58-4.81 B.M
Fe(III) Schiff base Complex	51.10 (51.08)	3.37 (3.35)	4.26 (4.24)	16.97 (16.95)	Light Gray	5.10-5.40 B.M
Mn(III) Schiff base Complex	51.24 (51.22)	3.38 (3.35)	4.27 (4.25)	16.74 (16.72)	Brown	4.81-5.62 B.M

Antibacterial Activity

Antibacterial activity was determined by Agar-ditch method. The investigated microorganisms were *Pseudomonas aeruginosa*, *Proteus vulgaris*, *Proteus mirabilis*, *Klebsiella pneumoniae* and *Staphylococcus aureus*. The metal complexes were dissolved in one of the two solvent 1, 4 di oxane (non polar) or DMF (polar) solvents to obtained final concentration 1mg/0.1ml. A loop full of the given test strain was inoculated in 30ml of nutrient broth and incubated for 24 hour in an incubator at 30°C in order to activate the bacterial strain activity. 18-20 ml of the

nutrient agar media was added in to a 100mm diameter pantry-plate. 0.1ml of the activated strain was inoculated in to the media when it reaches the temperature of 30°C. The medium was allowed to solidify. After solidification of the media a hole was made in the plates with the help of a cup-borer, which was then filled with one of the test sample solution. Controls were run (for each bacterial strain and each solvent), where pure solvent was inoculated in to the hole. The plates were incubated for 24 hours at 24°C. The inhibition zone formed by these compounds against the particular test bacterial strain determined

the antibacterial activity of the synthetic complexes. The mean value obtained for three individual replicates was used to calculate the zone of growth inhibition of each sample. The antibacterial activity of Schiff base metal complexes in DMF (polar) and 1, 4 dioxane (non polar) against *P. aeruginosa* is shown in

fig. -1 and fig. -2, respectively. All complexes showed greater activity in the polar solvent DMF, then the non polar solvent 1, 4 dioxane. In DMF Cr(III) complexes of Schiff base showed the best activity against all strains.

Fig-1 Antibacterial Activity of Metal Complexes in DMF

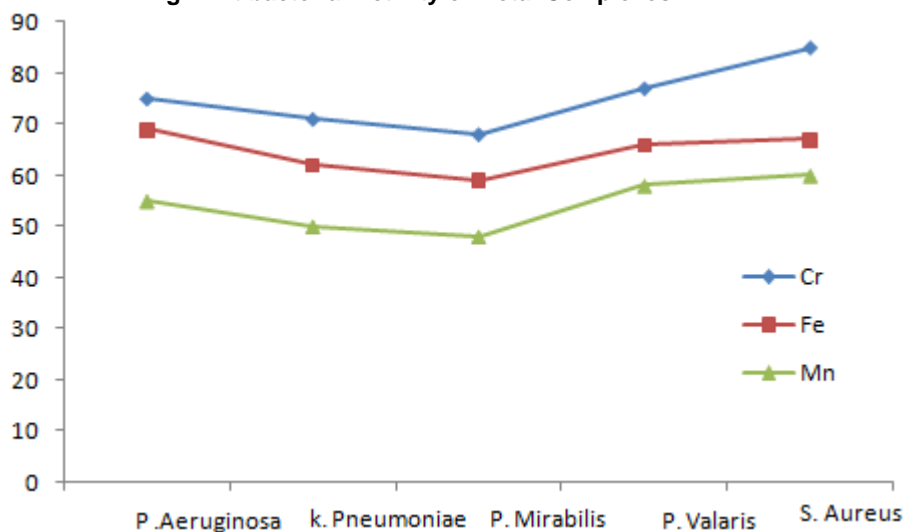
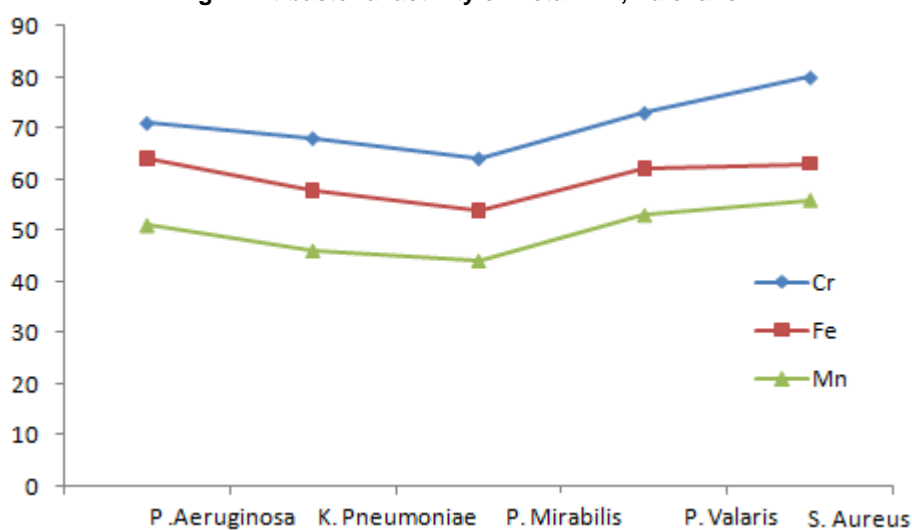


Fig-2. Antibacterial activity of Metal in 1,4-dioxane



Conclusion

For the above results, it can be concluded that amongst the four metals used for complexes formation, in non polar solvent 1, 4 dioxane Zn complex of Schiff base showed the best antibacterial activity but in polar solvent DMF, Cr (III) complex showed best antibacterial activity.

Endnotes

1. McKee T, McKee J. *Biochemistry*, Wm. C. Brown Publishers, Dubuque, 1996.
2. Pandeya SN, Sriram D, Nath G, De Clercq E., *Pharm. Acta Helv.* 74 (1999) 11.
3. Ranganatha VL, Prashanth T, Patil HBV, Bhadregowda DG, Mallikarjunaswamy C., *Chemical Data Collections* 13-14 (2018) 1.
10. Morad F M., El-ajaily M M. & Ben Gweirif, s, *Journal of Science and its applications*, 1, 1, (2007) 72.

4. Singh WM, Dash BC., *Pesticides*,. 22 (1988) 33.
5. Kelley JL, Linn JA, Bankston DD, Burchall CJ, Soroko FE, Cooper BR., *J. Med. Chem.* 38 (1995) 3676.
6. Turan-Zitouni G, Kaplancikli ZA, Özdemir A, Chevallet P., *Pharm. Chem. Life Sci.* 340 (2007) 586.
7. Tarafder MTH, Kasbollah A, Saravanan N, Crouse KA, Ali AM, OoKTOo., *J. Biochem. Mol. Biol. Biophys.* 6 (2002) 85.
8. Mruthyunjayaswamy B H M., Omkar B, Ijare&Jadegoud Y, *J. Braz. Chem. Soc.* 16, 4, (2005) 783
9. Maihub A.A., El-ajaily M.M., & Filog S. *Al-yarmousk Al-abhath journal*, 14, 1, (2005), 119.
11. Nair R., Scah A., Baluaj S., & Chanda, s, *J. Serb. Chem. Soc.* 71, 7, (2006), 733

12. Sawodny W, Riederer M, & Urban E, *Inorg. Chim. Acta*, 29, (1978), 63
13. Campos A, Anaconda J R, & Campos-Vaiiette M M, *Main Group Metal Chem*, 22 (1999) 283
14. El-ajaily M .M., Ben Gweirif, S., Maihub A.A.& El-tajoury A.N, *Science and its Application Journal*, 1, (2005), 196
15. Sonmez M. & Sekerel M, *Polish Journal of chemistry*, 76, (2002), 907
16. Sari N, Arslan S, Logoglu E & Sakryan I, *G. U. J. Sci.* 16 (2003) 283
17. Hassan A .M. A, *Journal of Islamic Academy Science*, 4, 4, (1991) 271
18. Toi N., Yin K., Chen Q. & Tan, *Molecules*, 8, (2005) 439
19. Bensaber S. M., Maihub A. A., Hudere S S & El-ajaily M. M, *Microchemical Journal*, 81, (2005), 191
20. Raman N., El-ayaily M .M. & Filoy S.M, *Alyamouk Al-abhath Journal*, 14,1, (2007) 119
21. Gunthkal MS, Goudal TR, Patil SA., *Oriental J. Chem.* 16 (2000) 151.
22. Kulkarni A, Patil SA, Badami PS. *Synthesis, Eur. J. Med. Chem.* 44 (2009) 2904.
23. Shakir M, Chingsubam P, Chishti HTN, Azim Y, Begum N., *Indian J. Chem.* 43A (2004) 556.
24. Niasari MS, Bazarganipour M, Ganjali MR, Norouzi P., *Transition Met. Chem.* 32 (2007) 9.
25. Wood JS., *Prog. Inorg. Chem.* 16 (1972) 227.
26. Kumar G, Devi S, Johari R., *E-Journal of Chemistry*. 9 (2012) 2255.
27. Figgis BN, Lewis J, *Prog. Inorg. Chem.* 6 (1965) 37.
28. Singh DP, Kumar R, Sharma C., *Eur. J. of Med. Chem.* 45 (2010) 1230.
29. Kumar G, Devi S, Kumar D, *Jou. of Mole. Str.* 1108 (2016) 680.