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RESEARCH ARTICLE

Synthesis and Characterization of Mixed Ligand Complexes of Cobalt

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Abstract: The synthesis and characterization of mixed ligand complexes of transition metal of I transition series such as Co is done by the condensation of Schiff base and 8-hydroxyquinoline. The synthesized complexes are characterized by using gravimetric analysis, CHNS, conductivity measurement and different spectral methods like IR and NMR.

Keywords: Transition metal, Schiff base, 8-Hydroxyquinoline, Conductivity measurement, IR and NMR

Introduction

The synthesis of mixed ligand complexes is very trendy now a days because of their ease of formation and also less time required to complete the reaction using ligands and metal salts[1]. These are the condensation product of primary amine and carbonyl compound. The common structural feature of these compounds is the azomethine group with a general formula $R_1HC=NR_2$, where R_1 and R_2 are alkyl, aryl, cyclo alkyl or heterocyclic groups which may be variously substituted[2,3].

8-Hydroxyquinoline has been used for many years for the preparation of mixed ligand complexes[4-7]. It forms stable chelate with metal in combination with some other ligands because of its ability to get bonded with metal with its phenolic oxygen and ring nitrogen[8-12].

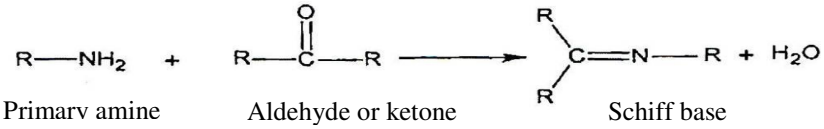
Experimental

All the chemicals such as salicyldehyde, benzaldehyde, 2-chlorobenzaldehyde, vanillin, 4-aminophenol, 8-hydroxyquinoline and metal chloride used are of good quality and are used without further purification.

Synthesis of Schiff base

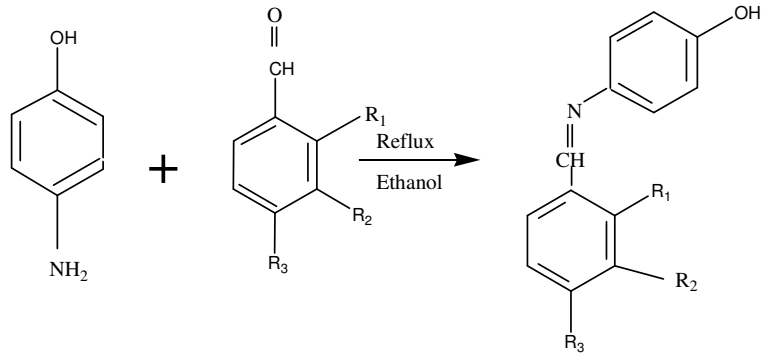
The Schiff base was prepared by condensing 2 g of amine taken in a round bottom flask containing 25 mL ethyl alcohol. Then 2 mL of aldehyde was added with continue stirring.

The mixture was refluxed for 2 hour. The Schiff base ligand was isolated by crystallization after volume reduction by concentrating in a china dish. The crystalline product was dried, collected and put in a desiccator till further use[13,14].



Primarv amine Aldehyde or ketone Schiff base

In the present investigation Schiff base ligands were prepared by the condensation of 4-aminophenol with 2-chloro-benzyldehyde, salicylaldehyde, vanillin and benzaldehyde (Scheme 1).



R_1: H, OH, Cl, R_2: H, OCH_3, R_3: H, OH, Cl

Scheme 1.

Synthesis of metal complex

Hot methanolic solution (20 mL) of 8-hydroxyquinoline (0.05 mmol) was added to the hot methanolic solution (20 mL) of Schiff base (0.05 mmol) in two round bottom flasks. Now the metal chloride solution (10 mL, 0.05 mmol) in methanol was added drop wise to the above reaction mixture. The resulting reaction mixture was refluxed on a magnetic stirrer for about 6 h. The progress of the reaction was checked by thin layer chromatographic technique using solvent system 8:2 (Pet-ether+Ethyl acetate). After 6 h refluxing was stopped and the reaction mixture was allowed to cool for about an hour at room temperature. The dark coloured precipitate appeared, then filtered, collected and allowed to dry *in vaccum* desiccators.

Results and Discussion

Schiff base

The physical data of Schiff base having general formula $R_1N{=}CHR_2$ has been shown in the Table 1.

Metal complexes

All the mixed ligand complexes synthesized are coloured. They have strong metal-ligand bond indicating thermal stability. These complexes are synthesized using molar conductivity, thin layer chromatography, solubility and spectral data.

Molar conductance measurements

Molar conductance values for all the synthesized complexes were measured in DMSO (10^{-3} M) at room temperature. The value of molar conductivity lies in the range of 15-21 mho cm^2 mol^{-1}

which is much less than the value of 70-160 mho cm^2 mol^{-1} obtained for 1:1 electrolyte in the solvent. Thus, it can be concluded that these complexes are non-electrolytic in nature.

Table 1. Physical data of Schiff base

R_1	R_2	Molecular formula	Melting point ^oC	Colour	Solubility
4-Aminophenol	Salicyldehyde	$C_{13}H_{11}NO_2$	141-154	Reddish crystalline solid	C_2H_5OH, DMSO,$CHCL_3$
4-Aminophenol	Vanilin	$C_{14}H_{12}ClNO_2$	207-215	Brown crystalline solid	C_2H_5OH, DMSO
4-Aminophenol	2-Chlorobenzaldehyde	$C_{13}H_{10}ClNO$	149-158	Brown Solid	C_2H_5OH, DMSO
4-Aminophenol	Benzaldehyde	$C_{13}H_{11}NO$	182-142	Pale Yellow Crystalline Solid	C_2H_5OH, DMSO

Thin layer chromatography

The TLC was performed by using silica gel in polar solvent pet-ether and ethyl acetate (20 %). The spray reagent used was iodine vapours. At the end the starting material disappeared and all the complexes appeared as a single spot.

Solubility

The solubility of mixed ligand complexes along with 8-hyroxyquinoline was checked with different solvent. The complexes were partly soluble in EtOH and MeOH, insoluble in acetone and water, highly soluble in DMF and DMSO.

Infrared spectra

The important infrared spectral bands and their assignments for the synthesized ligands and complexes were recorded as KBr discs. The IR spectra for free ligands and its metal complexes were recorded within the IR range 4000-400 nm. An important feature of infrared spectra of the metal complexes with 8-hydroxyquinoline is the absence of band at 3440 cm^{-1} due to the O-H stretching vibration of the free O-H group of hydroxyquinoline[15]. This observation leads to the conclusion that complex formation takes place by the deprotonation of hydroxyl group of the hydroxyquinoline moiety[16], reported that for several metal complexes with HQ, V(C-O) band is observed at 1120 cm^{-1}. The position of this band undergoes variation depending on the metal complex under study. A strong v(C-O) band is observed in the range 1103-1112 cm^{-1} indicating the presence of oxine moiety in the complexes coordinating through its nitrogen and oxygen atoms as uninegative bidentate ligand. The v(C=N) mode in oxine occurs at 1499-1502 cm^{-1} in the spectra of metal complexes. This band is observed in the spectrum of the ligand in the higher region (1580 cm^{-1}). A negative shift in this vibrational mode on complexation indicates the coordination through tertiary nitrogen donor of HQ. The in plane and out of plane ring deformation modes are observed at 505 and 787 cm^{-1} respectively, confirming coordination through the nitrogen atom of HQ with metal. The M-O stretching is observed in the range of 508-599 cm^{-1}

H[1] NMR spectra

The H^1 NMR spectra of ligands and their respective complexes in DMSO solution were also compared. The free NH_2 protons usually show a broad singlet peak in a region of 4-6 ppm. This signal is absent in the observed spectra of Schiff bases which indicates the formation of Schiff bases. The peaks for aromatic proton exhibits signals in the region 6.06-7.42 ppm. The ^{1}H NMR spectra of all the complexes exhibits signals at 10.26 and 10.35 ppm due to CH=N- group as shown below in Figure 1.

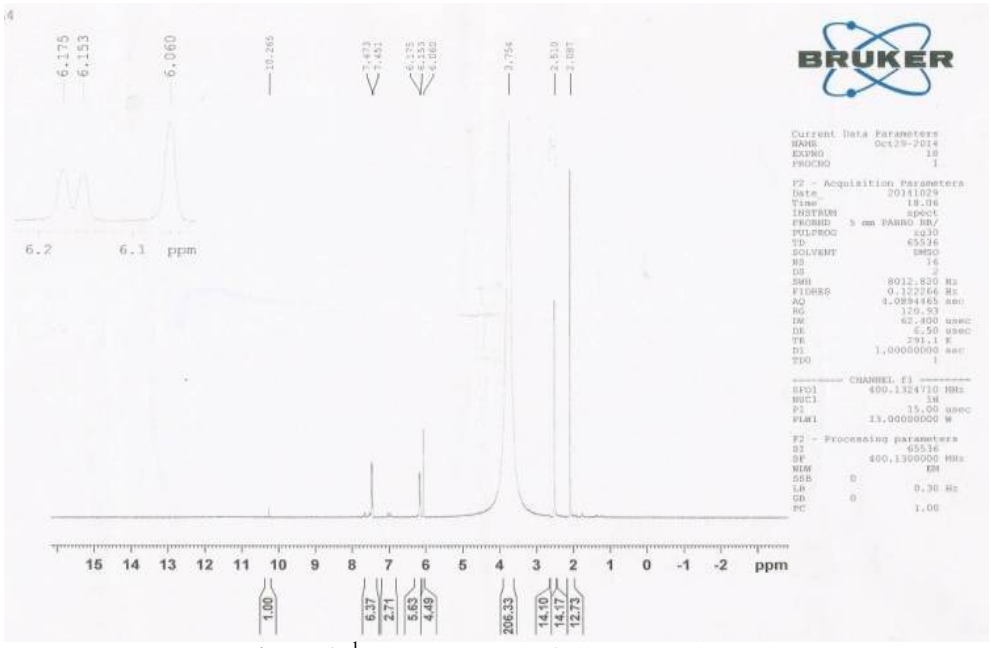


Figure 1. ^{1}H NMR spectra of all the complexes

Antibacterial activity

The bacterial culture for the bacteria namely, *M. luteus*, *E. faecalis*, *A. dinitroficans* and *K. pneumonia* was grown on nutrient agar medium. In each plate the central well was filled in with standard antibiotic Chloramphenicol and two well on the edges are of Schiff base complexes. From the result obtained, it has been found that the tested complexes show activity against *E. faecalis* and *A. Dinitroficans* and show a little activity against *A. Luteus* and *K. Pneumonia* Figure 2 and 3.

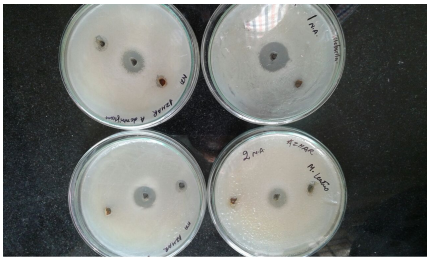


Figure 2. Complexes showing more activity against *E. faecalis* and *A. Dinitrificans*

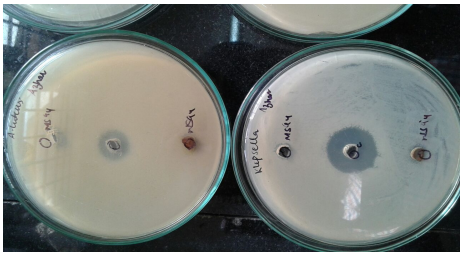


Figure 3. Complexes showing less activity against *A. Luteus* and *K. Pneumonia*

Conclusion

The complexes are obtained as coloured powdered materials and are characterized using IR spectra and ^{1}H NMR. The complexes are completely soluble in DMF and DMSO.

References

1. Kiruthika M, Elayaperumal R and Vennila T, *Der Chemica Sinica*, 2012, **3(5),** 1175-1180.
2. Valcarcel M and Laque de Castro M D, Flow-Throgh Biochemical Sensors, Elsevier, 1994, Amsterdam.
3. Worku D, Negussie M, Raju V J T, Theodros S, Jonsson J A, *Bull Chem Soc., Ethiop*., 2002, 29
4. Anjaneyulu Y, Swamy R Y and Prabhakara Rao R, *Proc Indian Acad Sci* (*Chem. Sci.),* 1984, **93(2),** 131-138.
5. Thirumalaikumar M, Sivakolunthu S, Ponnuswamy A and Sivasubramanian S, *Indian J Chem.,* 1999, **38A**, 720-722.
6. Shivshankar V S and Takkar N V, *Acta Poloniae Pharmaceutica-Drug Res.*, 2003, **60(1),** 45-50.
7. Shivshankar V S and Takkar N V, *Acta Poloniae Pharmaceutica-Drug Res.*, 2004, **61(2),** 127-133.
8. Alazawi S A S and Alhamadani A A S, *Um-Salama Science Journal*, 2007, **4(1),** 102-109.
9. Dubey R K, Dubey U K and Mishra C M, *Indian J Chem*., 2008, **47A**, 1208-1212.
10. Halli M B, Patil V B, Sumathi R B and Mallikarjun K, *Der Pharma Chemica*, 2012, **4(6),** 2360-2367
11. Usharani M, Akila E and Rajavel R, *J Chem Pharm Res*., 2012, **4(1),** 726-731.
12. Mohamed G G and Abd El-Wahab Z H, *Spectrochim Acta Part A: Mole Biomolecular Spectroscopy*, 2005**, 61(6),** 1059-1068; DOI:10.1016/j.saa.2004.06.021
13. Schiff's H, *Ann Chem*., 1864, **3(Suppl),** 343
14. Tomonori Ohba, Hirofumi Kanoh and Katsumi Kaneko, *J Am Chem Soc*., 2004, **126(5),** 1560-1562; DOI:10.1021/ja038842w
15. Nakamoto K, Morimoto Y and Martell A E, *J Am Chem Soc*., 1961, **83,** 4528-4532; DOI:10.1021/ja01483a009
16. Charles R C, Freiser H, Friedel R, Hillard L E and Johnson W D, *Spectrochimica Acta*, 1956, **8(1)**, 1-8; DOI:10.1016/0371-1951(56)80018-0