



SYNTHESIS, CHARACTERIZATION AND BACTERIAL SCREENING TRANSITIONAL METAL COMPLEXES TRIDENTATE SCHIFF BASES OF BENZENEDIAMINE

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Abstract

The Schiff base of various substituted Benzene diamine derivatives were synthesized from benzene 1,2 diamine and o-substituted Benzaldehyde in ethanolic media with maintaining appropriate stoichiometry of precursors. The metal complexes also synthesized with transitional metal ion such as Ru^{3+} , Mo^{3+} , Rh^{3+} , Zn^{2+} and Cr^{3+} . The Schiff bases were analyzed by physical tools such as NMR, IR and UV and Mass Spectroscopy. The electronic spectra of complex exhibit existence of $\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$ ($>C=N$). All the complexes show the moderate antibacterial activity.

Keyword

Schiff base complex, 2-nitrobenzaldehyde, 2-Chlorobenzaldehyde, 2-hydroxy benzaldehyde, 2-methoxybenzaldehyde, benzene 1,2 diamine, antibacterial activity.

1. Introduction-

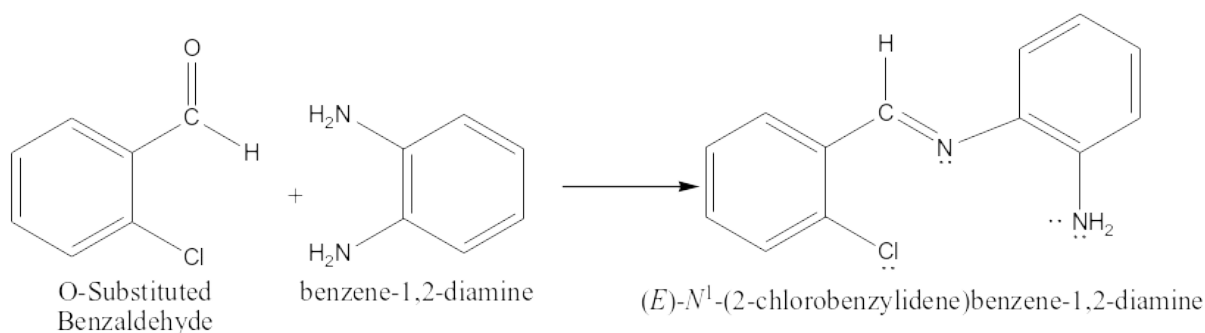
Co-ordination Chemistry is undoubtedly the most active research area in inorganic chemistry. The number of coordination complex compounds have been synthesized and analyzed during the last decades for its biological importance, Physiological, Industrial utilization for human kind. The Schiff base of 2-nitrobenzaldehyde, 2-chlorobenzaldehyde, 2-hydroxybenzaldehyde, 2-methoxybenzaldehyde with benzene 1,2 diamine was added by stoichiometric proportionality in ethanolic media with 2 hours by refluxing in round bottom flask at 90-120°C. The resulting solution was evaporated under vacuum to remove the solvent. The product was collected by filtration washed several times with ethanol and recrystallized from hot ethanol and dried under vacuum. The yellow product was obtained.

2. Experimental-

Chemicals –The entire chemicals used for precursor material for synthesis of Schiff base and their complexes were A.R. Grade and solvent were purified double distillation and by molecular sieve Molecular sieves, Grade 513 (Type 4A; 4-8 Mesh)for this work.

3.Synthesis Schiff bases-The Schiff bases from ortho substituted Benzaldehyde and benzene 1,2 diamine was prepared by adding equimolar quantities ortho- substituted benzaldehyde ethanolic solution to same volume of ethanolic solution benzene 1,2 diamine. The mixture was stirred for two to four hours. The resulting solution was evaporating under vacuum to remove the solvent. The product was collected by filtration, washed several times with ethanol and recrystallised from hot ethanol and then dried under vacuum. The colour of product was yellow.

Synthesis of tridentate Schiff base-

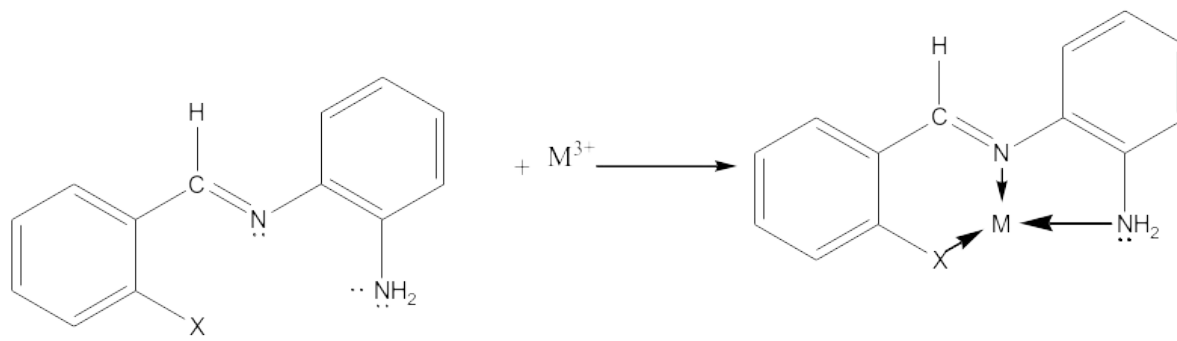


Where- X(-NO₂, -Cl, -OH, -OMe)

4.Synthesis of Metal ligand complexes –

The mixture of Schiff's bases with (in equimolar) under investigation in 25 cm³ ethanol and same amount of same solvent of M (III) salt was reflux for two hours. The pale yellow crystal was collected by filtration then washed by several times with hot ethanol until washing becomes colorless. The product was dried in air and stored in desiccators over anhydrous CaCl₂ under vacuum. A better yield 90% was obtained. The purity of complex was confirmed by elemental analysis.

Synthesis of Metal-ligand Complexes -



(E)-2-(2-chlorobenzylideneamino)phenol

Where M- Ru³⁺, Mo³⁺, Rh³⁺,
 Zn²⁺, Cr³⁺

5.Result and Discussion-

1. Characterization-

- a) ¹H NMR –Protocol of ¹H NMR of Schiff base shows benzylidenimineCH 7.17, NH₂(4.00 aromatic C-NHH,) 8.39, 8.11 benzylidenimin, CH 6.5 , 7.26 for 2 second-benzene.

b) Elemental analysis -

The elemental analysis for carbon, hydrogen and nitrogen were performed by micro-analytical method. IR spectra of ligand and its complex were carried out by using KBr disc IFS-25 DPUS/IR spectrometer. The electronic absorption spectrum was carried out using Perkin-Elmer Lambda 4β spectrophotometer in 1cm matched silica cell using CHCl₃ as a solvent. The molar conductance was carried out in DMSO using digital conductivity meter (ELICO)

c) Infrared Spectra of Schiff bases and its complexes-

The IR data of Schiff base and its complex with Ru (III) and Rh(III) ions are agreement with expected range. The peak at 1539cm⁻¹ shows due to C=N vibration. The band at 3450cm⁻¹



¹indicates H₂O molecule. The shifting of this group to lower frequency compared with free Schiff base 1530cm⁻¹ suggest the co-ordination of Ru³⁺, Mo³⁺, Rh³⁺, ion through nitrogen atom. The band at 1610cm⁻¹ shows C=O stretching frequency. The band at 3300cm⁻¹ -OH group in the complex.

d. Magnetic moments-

The magnetic moment value of the complex revealed that existence of diamagnetic characters. The obtained data confirmed square planar geometry around the Zn(II) and octahedral geometry of Ru (III), Mo (III), Rh (III), Cr (III) complex.

e. Antibacterial activity of Schiff base complexes-

Antibacterial activity of Schiff base complexes was tested on gram + ve bacteria Staphylococcus aureus, gram -ve bacteria Pseudomonas and E.coli. The antibacterial activity of sample was evaluated by measuring the inhibition observed around tested material.

6. Acknowledgement-

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References -

1. Panneerselvam P, Nair RR, Vijayalakshmi G, Subramanian EH, Sridhar SK. Synthesis of Schiff bases of 4-(4-aminophenyl)-
2. Morpholine as potential antimicrobial agents. Eur J Med Chem 2005;40(2):225–9. [22] Dalpozzo R, de Nino A, Nardi M, Russo B, Procopio A. Erbium(III) triflate: a valuable catalyst for the synthesis of aldimines, ketimines and enamines. Synthesis 2006;7:1127–32.
3. Naeimi H, Salimi F, Rabiei K. Mild and convenient one pot synthesis of Schiff bases in the presence of P₂O₅/Al₂O₃ as new catalyst under solvent-free conditions. J Mol Catal A Chem 2006;260(1–2):100–4.
4. Gopalakrishnan M, Sureshkumar P, Kanagarajan V, Thanusu J, Govindaraju R. Silica gel supported sodium hydrogen sulfates an efficient and reusable heterogeneous



catalyst for the synthesis of imines in solvent-free conditions under microwave irradiation. *J Chem Res* 2005;5:299–303.

5. Gopalakrishnan M, Sureshkumar P, Kanagarajan V, Thanusu J. New environmentally-friendly solvent-free synthesis of imines using calcium oxide under microwave irradiation. *Res Chem Intermed* 2007;33(6):541–8.
6. Guzen KP, Guarezemini AS, O' rfa~o ATG, Cella R, Pereira CMP, Stefani HA. Eco-friendly synthesis of imines by ultrasound irradiation. *Tetrahedron Lett* 2007;48(10):1845–8.
7. Gedye R, Smith F, Westaway K, Ali H, Baldisera L, Laberge L, et al. The use of microwave ovens for rapid organic synthesis. *Tetrahedron Lett* 1986;27(3):279–82.
8. Giguere RJ, Bray TL, Duncan SM, Majetich G. Application of commercial microwave ovens to organic synthesis. *Tetrahedron Lett* 1986;27(41):4945–8.