

ORIGINAL ARTICLE

Spectral characterization and biological evaluation of Schiff bases and their mixed ligand metal complexes derived from 4,6-diacetylresorcinol



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Abstract In the present study two new series of Copper(II), Nickel(II) and Cobalt(II) complexes with two newly synthesized Schiff base ligands 4,6-bis(1-(4-bromophenylimino)ethyl)benzene-1,3-diol (H_2L^1), 4,6-bis(1-(4-methoxyphenylimino) ethyl)benzene-1,3-diol (H_2L^2) and organic ligands 8-hydroxy quinoline, 1,10-phenanthroline have been prepared. The Schiff bases H_2L^1 and H_2L^2 ligands were synthesized by the condensation of 4,6-diacetyl resorcinol with 4-bromo aniline and 4-methoxy aniline. The ligands and their metal complexes have been characterized by FT-IR, Mass, 1H NMR, UV-Vis., elemental analysis, ESR and Thermal gravimetric analysis. The Schiff base and their metal complexes were tested for antimicrobial activity against gram positive bacteria *Staphylococcus aureus*, *Streptococcus pyogenes* and gram negative bacteria *Escherichia coli*, *Pseudomonas aeruginosa* and fungus *Candida albicans*, *Aspergillus niger* and *Aspergillus clavatus* using Broth Dilution Method.

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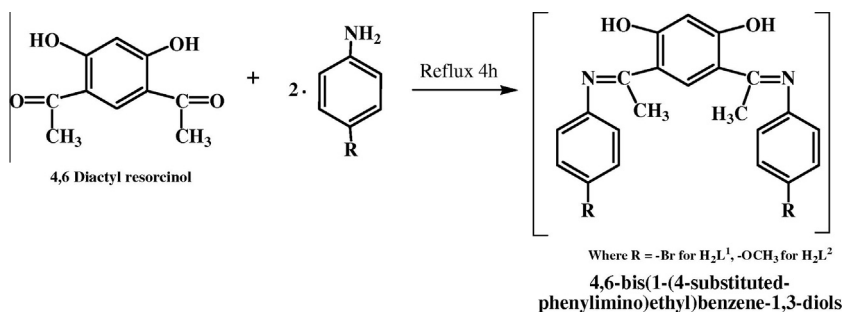
1. Introduction

Metal chelation is involved in many important biological processes where the coordination can occur between a variety of the metal ions and a wide range of ligands (Rosette, 2002; Crichton, 2008). Generally, the chelating ligand is a polyfunctional molecule which can encase the metal in an organic sphere. Many types of the Schiff base ligands are known and the properties of their derived metal chelates have been investigated (Pandya and Ganatra, 2008; Pandya and Shah, 2008). Acyclic ligands having nitrogen, oxygen and sulfur donor atoms in their structures, can act as good chelating agents for transition and non-transition metal ions (Muhammad et al., 2007; Spinu et al., 2008). Numerous Schiff bases and

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Scheme 1 Reaction scheme of ligand.

their transition metal complexes have been investigated by various techniques for different purposes (Parmar and Teraiya, 2009; Sitkowski et al., 1996; Tanaka et al., 1991; Vyas et al., 2011a). Schiff base metal complexes have been widely studied because they have industrial, antifungal and biological applications (Kumar and Chandra, 2011; Offiong et al., 2000; Patil and Chaurasiya, 2008). They serve as models for biologically important species and find applications in biomimetic catalytic reactions. Chelating ligands containing O and N donor atoms show broad biological activity and are of special interest because of the variety of ways in which they are bonded to metal ions (Hung and Lin, 2009). Alkyl resorcinol and aromatic resorcinol are reported to possess valuable therapeutic and anti-septic properties. Alkyl resorcinol like 4-n-butyl resorcinol has been used in skin creams and lotions which are claimed to have good bleaching and antimicrobial effects. 2-Alkyl resorcinol (where the alkyl group is linear) has been reported to have skin depigmentation properties (Gadgil et al., 2004; Bolinger et al., 1990). 8-Hydroxyquinoline and 1,10-Phenanthroline have been extensively used as a ligand in both analytical and preparative coordination chemistry. As an important building block, both the ligands units play an important role in the development of the supramolecular chemistry (Li et al., 2007).

2. Experimental

2.1. Materials

4,6-Diacetylresorcinol was synthesized according to the method reported in the literature (Anjaneyulu et al., 1979). Copper(II), nickel(II) and cobalt (II) were used as nitrate salts and were obtained from rankem. All amines and organic ligands (8-hydroxyquinoline and 1,10-phenanthroline) were used from Merck, organic solvents EtOH, MeOH, DMF and DMSO were reagent grade.

2.2. Physical measurements

IR spectra (4000–400 cm^{-1}) of the metal chelates were obtained using KBr discs, on 8400 FT-IR SHIMADZU spectrometer. Mass spectra were recorded on QP 2010 SHIMADZU GCMS spectrometer. ^1H NMR spectra of ligands were recorded on Bruker Avance II 400 MHz FT-NMR spectrometer using TMS as an internal standard and DMSO- d_6 as a solvent. ESI mass spectra of complexes were recorded VG-70-S Spectrometer. Electronic spectra of the metal

complexes in DMF were recorded on a Perkin Elmer Lambda 19 spectrophotometer, and ESR was recorded on E-112 ESR spectrometer, at X-band microwave frequency (9.5 GHz) with sensitivity of 5×10^{10} ΔH spins. Molar conductance of the metal complexes was determined on Systronics direct reading conductivity meter type CM-82T. TGA was carried out by using Perkin Elmer (Pyris 1 TGA) from 50 $^\circ\text{C}$ to 800 $^\circ\text{C}$ under heating rate of 10 $^\circ\text{C}/\text{min}$. Elemental analysis (C, H and N) were carried out on Elemental Analyzer PERKIN ELMER 2400, and analysis of metal was carried out by EDTA titration method, in which the metal complex first evaporate in conc. nitric acid and prepare a stock solution. This solution with ammonia then titrates against EDTA by using appropriate indicator. The M.P. of ligands was carried out by a standard laboratory thermometer. Magnetic moment of the compound was measured by GOUY balance using $\text{Hg}[\text{Co}(\text{CNS})]$ as standard.

2.3. Synthesis of Schiff bases

The Schiff base ligands H_2L^1 and H_2L^2 were synthesized by adding 4,6-diacetyl resorcinol (4.85 g 25 mmol) dissolved in hot absolute EtOH (20 cm^3) to 4-bromoaniline (8.6 g, 50.0 mmol) and 4-methoxy aniline (6.15 g, 50.0 mmol) respectively, in absolute EtOH (20 cm^3). The reaction mixtures were heated to reflux for 4 h. The products obtained were filtered off and washed several times with a small amount of EtOH then ether. The products were kept in a desiccator until used. Recrystallization was carried out in EtOH. The progress of the reaction was monitored by TLC. The yields were 85% for H_2L^1 and 80% for H_2L^2 . M.P. of these two ligands are 179 $^\circ\text{C}$ and 192 $^\circ\text{C}$, respectively (Scheme 1).

2.4. Synthesis of metal complexes

An ethanolic solution of the metal (II) nitrate (hexahydrate) (20 ml) was gradually added to an ethanolic (30 ml) solution of the Schiff base ligand in 2:1 molar ratio and the solution was stirred for 1 h. An ethanolic solution of the other ligands L' ($L' = 8\text{-HQ}$, 1,10-Phen) was then added to the previous solution in the molar ratio 2:1. The solution was continuously stirred for 4 h, during which the metal complex precipitated. The resulting precipitates were filtered off, washed with ethanol then diethyl ether and finally air-dried. The complex is air stable in the solid state and soluble in DMF and/or DMSO. The progress of reaction was monitored by TLC (Scheme 2).

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