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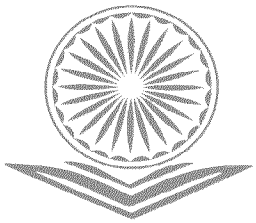

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8. Synthesis and Biological Evaluation of Metal Complexes of O, N/S Donor Ligand Derived from Dehydroacetic Acid

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Abstract

The solid metal complexes of Cu (II), Ni (II), Fe (III), Co (II), Mn (II) and Cd(II) ion with O,N/S donor ligand derived from Dehydroacetic Acid and 2-amino-6-picoline were synthesized, characterized by elemental analysis, (FTIR and ^{1}HNMR) spectroscopic techniques. The biological (Anti-bacterial, Ant-fungal and Antioxidant) activity of ligand and metal complexes have been screened in vitro against 1) *B. Subtilis* 2) *S. aureus* 3) *A. niger* and 4) *C. albicans*. The composition and structure of synthesized O, N/S donor ligand and all metal complexes confirmed by elemental analysis, (FTIR and ^{1}HNMR) spectroscopic techniques. The synthesized Schiff base ligand and all metal complexes are found to be biologically active. Co (II) and Cu (II) complexes exhibit maximum and Fe (III) complex exhibits minimum zone of inhibition. Cd (II) and Mn (II) complexes were found to be Best Radical scavengers.

Keywords: Dehydroacetic Acid, O, N/S donor ligand, Metal Complexes, Biological activity.

Introduction

Schiff bases and their metal complexes can catalyze the reactions, show biological activities such as antimicrobial, antifungal, antiviral, synergistic, antioxidant, anti-inflammatory, analgesic, antitumor, cytotoxic, antidiabetic, anti-fertility[1,2], anti-tumor activity[3] DNA Photoclevage activity [4] etc. In the 21st century, coordination Chemistry becomes the rapid developing branch of chemistry due to large applications in the field of organic synthesis, industries, dyes, drugs and as bleaching agents. The metal complexes are applied in medicinal, analytical and diagnostic purposes in the living system.

Schiff bases and their metal complexes can catalyze the reactions, show biological activities such as antimicrobial, antifungal, antiviral, synergistic, antioxidant, anti-inflammatory, analgesic, antitumor, cytotoxic, antidiabetic, anti-fertility[1,2], anti-tumor activity[3] DNA

Photoclevage activity [4] etc.Popova and Berova reported that copper complexes are good for a liver function and its level in blood and urine has an influence in a pregnancy disorder, Nephritis Hepatitis, and Anemia in children[5] Leukemia, Leprosy.

In this work we describe the synthesis and characterization of the Schiff base derived from DHA and aromatic primary amines (2-amino-6-picoline) and metal complexes. The biological activities of ligand and metal complexes have been investigated.

Experimental Details

Dehydroacetic acid, 2-amino-6-picoline and metal chlorides are procured from E-Merck Germany, Avra Pvt. Ltd. and Loba chemie Pvt. Ltd. The solvents were dried and distilled before use by following procedure[6]. The IR spectra of ligand and metal complexes were recorded on Perkin Elmer (1430) FTIR spectrophotometer. The ^{1}HNMR spectrum of ligand is recorded on Brucker FT-500 MHz NMR Spectrophotometer in $CDCl_3$ by using solvent and TMS as reference substance.

Synthesis of O, N/S Donor Ligand

The O, N/S donor ligand is synthesized by using standard procedure.[7,8]

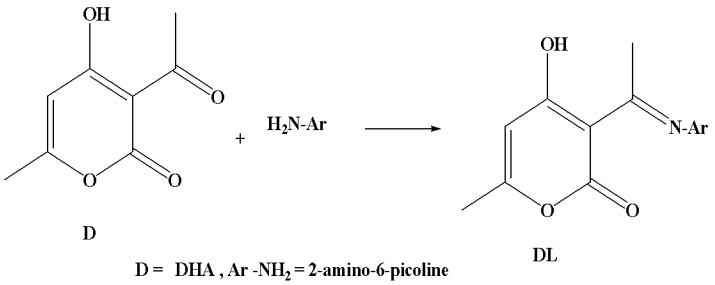

Fig.01: O, N/S Donor Ligand

Synthesis of Metal Complexes

Transition metal complexes are synthesized by using standard procedure[8].

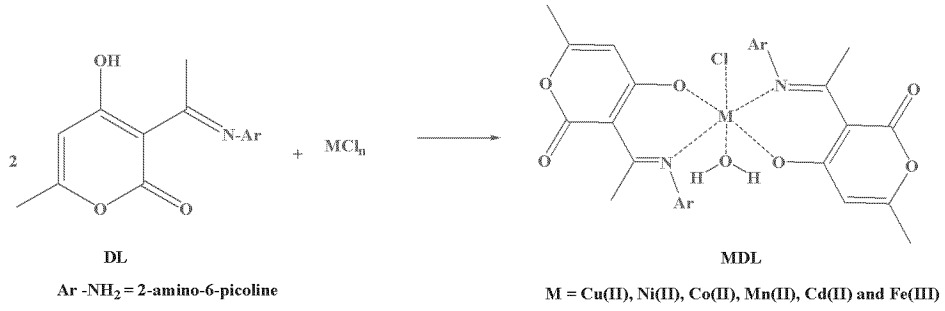

Fig.02: Synthesis of Metal Complexes

Results and Discussion

Synthesized O, N/S donor ligand is yellow colored solid stable to air, non-hygroscopic, insoluble in water and soluble in hot alcohols. All the metal complexes were colored solids, stable to air non-hygroscopic. They were insoluble in water, alcohol, but soluble in DMF and DMSO. The melting points of ligand and metal complexes were determined by open capillaries.

^{1}H-NMR

The ^{1}HNMR spectra of O,N/S donor ligand in $CDCl_3$ at laboratory temperature showed the signals at δ (ppm) values 2.2 (3H, s, $C_6 - CH_3$) , 15.82 (1H, s ,O-H) , 5.75 (1H, s, C_5-H) , 2.58 (3H, s, N=C-CH_3), for Dehydroacetic acid moiety. 7.5- 7.8 (3H, m, Ar. C_3, C_4 & C_5 H), 2.3 (6H, Ar-CH_3) of picoline moiety.

IR Spectra

The IR spectrum of free O, N/S donor ligand (DL) showed a broad weak band 3432 cm^{-1} appear due to ν (O-H--) stretching .The band at 1698cm^{-1} assigned to ν (C=O) stretching of lactone carbonyl, 1656 cm^{-1} ν (C=N) stretching of azomethine, 1342 cm^{-1} ν (C-N) stretching of aryl azomethine & 1260 cm^{-1} ν (C-O) stretching of enolic group.

The disappearance of broad weak band at 3432 cm^{-1} in the spectra of all metal complexes suggest the deprotonation enolic oxygen, it is supported by upward shift in ν (C-O). The azomethine N in to coordination to the metal ion is further supported by downward shift in ν (C=N) and upward shift in ν(C-N). This indicates the participation of azomethine N and enolic O in complexation.

The IR Spectra of the metal complexes showed new bands in the region of 637-554 & 485-459 which can be assigned to ν (M-O) & ν (M-N) vibrations respectively.[9,10].

Antibacterial and Antifungal Activity

The antimicrobial potentiality of DHA Schiff base ligands and their metal complexes was carried out by the agar well diffusion method[11]. The minimum inhibitory concentration of O,N/S donor ligand and their metal complexes was determined by adopting the standard procedure of the National Committee for Clinical Laboratory standard (2004)[12]. In vitro antibacterial and antifungal activity was screened by considering zone of inhibition of growth. The synthesized O, N/S donor ligand DL and its metal complexes were screened with their different concentrations with standard antibiotics such as streptomycin and griseofluvin (1 mg/mL).

O,N/S donor ligand weak anti bacterial but shows strong antifungal activity. Antibacterial activity significantly increases on coordination because coordination reduces the polarity of metal ion due to partial sharing of positive charge with ligands that increase the lipophilic nature of metal ion in complex.[4,13] This enhanced antimicrobial activity of complexes is because of the lipophilic nature of metal ion in complex[14]. The Antimicrobial activity data is presented in **Fig.3.**

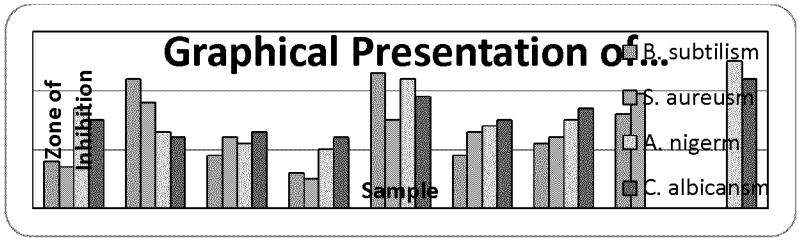

Fig.03: Graphical presentation of antibacterial and antifungal activities.

Antioxidant Activity

Antioxidant activity (Free radical scavenging activity) of O, N/S donor ligand derived from Dehydroacetic acid with 2-amino -6-picoline and their metal complexes was studied by using 1,1-diphenyl -2-picryl hydrazyl (DPPH) radical by standard procedure.[15,16] 50 -75 µM concentration solution of ligands and metal complexes in DMSO was added in to equal volume of DPPH. The reaction mixture was allowed to stand for 30 minutes at room temperature and then absorbance (O.D.) of the reaction mixture, solvent and vitamin C as standard was recorded at 540 nm on UV-Visible spectrophotometer. The scavenging activity was determined by measuring the reduced O.D. of DPPH. The antioxidant activity /Free radical scavenging activity of DHA Schiff base ligands and their metal complexes were performed with three replicates to obtain + S. D. (standard deviation).[17] Volume of solution used for each test is 50 µL in DSMO.

$$\% \text{ of Radical Scavenging Activity} = \frac{\text{Mean OD}_{\text{Standard}} - \text{Mean OD}_{\text{Sample}}}{\text{Mean OD}_{\text{Standard}}} \text{ X } 100$$

Antioxidant activity data is presented in **Fig. 4**

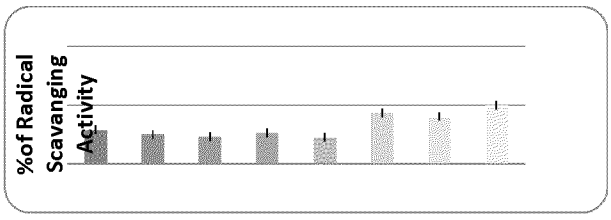

Fig 4: Graphical presentation of antioxidant activity of ligand and their metal complexes.

The O,N/S donor ligand and all-metal complexes exhibited DPPH radical scavenging activity. In accordance with the percentage Radical Scavenging activity, the compounds were categorized as Best radical scavengers and Poor radical scavengers. Ligand DL and complexes with Cd and Mn complexes were found to be Best Radical scavengers while the complexes of ligands L with Cu, Ni, Fe, and Co are proved to be Poor Radical scavengers.

Conclusion

Composition of O,N/S donor ligand and metal complexes confirmed by elemental analysis and structure of O, N/S donor ligand and metal complexes confirmed by IR and ^{1}H-NMR study. O,N/S donor ligand exhibits weak activity against bacteria but shows strong antifungal activity. Antibacterial activity significantly increases on coordination due to increase in lipophilic nature of metal ion in complex. Co (II) and Cu (II) complexes exhibited maximum zone of inhibition while Fe (III) complex exhibited minimum zone of inhibition. Cd and Mn complexes were found to be Best Radical scavengers while the complexes of ligands DL with Cu, Ni, Fe, and Co are proved to be Poor Radical scavengers.

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