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Research Article

SYNTHESIS, CHARACTERIZATION AND BIOLOGICAL STUDIES OF NI(II) AND ZN(II) COMPLEXES WITH N,N' –BIS(BENZOIN)-1,4 BUTANE DIIMINE

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ABSTRACT

Objective: The objective of the present study is to synthesize Schiff base ligand, N,N' –bis(benzoin) 1,4 butane diimine and its Ni(II) and Zn(II) complexes.

Methods: The synthesized complexes were characterized by IR and SEM. The synthesized Schiff base ligand and its Ni(II) and Zn(II) complexes were screened for antibacterial and antifungal activity.

Results: IR data show that the complexes are four coordinate. The SEM show that all the complexes are nano crystalline.

Conclusion: The synthesized complexes were tested against the bacterial and fungal species.The synthesized complexes were showed remarkable zone of inhibition with standards and have good antibacterial and antifungal activities.

Keywords: Schiff base , N,N' –bis(benzoin) 1,4 butane diimine, Metal Complex, Antimicrobial

INTRODUCTION

The reaction of primary amines and an aldehyde or a ketone forms a Schiff base. A Schiff base is a chemical compound containing of a -C=N- group [1-3]. Transition metal Schiff base complexes have been generally prepared by refluxing the solutions of a metal salt, a diamine and a compound containing a carbonyl group and a primary amine. Holm *et al.* (1966) reported the synthesis and characterization of some divalent copper complex compounds by refluxing the solutions of copper (II) salt, salicylaldehyde but different diamines. Thakur *et al*. [4] reported the synthesis and characterization of Ni (II), Co (II) and Fe (II) Schiff base complex compounds. The compounds were prepared by refluxing mixture of the solutions of a divalent metal chloride, acetylacetone and hydrazine hydrate.

However, recent reports indicated synthetic modification in which the ligand is separately prepared before its interaction with a metal salt. Xishi *et al*., 2003[5] showed the synthesis and spectroscopic properties of Mn (II), Co (II) and Cu (II) complexes with novel Schiff base ligand derived from 2, 2' bis(p-methoxylphenylamine) and salicylaldehyde. Schiff base complexes of transition metals are of great importance in medicine, biochemistry and industries among others. For example, the field of medicine has witnessed an increase in the number of complexes with therapeutic value. Cobalt (II) Schiff base complexes are potential antiviral agents, cis-dichlorodiamine platinum (II) complex is an anti cancer agent while copper (II) Schiff base is an anti-tubercular agent[6,7] .

This paper reports the synthesis and characterization of N, N' –Bis(Benzoin)- 1,4 butane diimine metal(II) complex compounds due to paucity of information.

MATERIAL AND METHOD

The chemicals and solvents used in this work were of Analar grade. All the glass wares used were washed thoroughly with distilled water and dried in an oven. Infrared spectra were recorded on a Perkin-Elmer FT-IR type 1650 spectrophotometer in wave number region 200-4000 cm^{-1}. SEM images were recorded in VEGA3 TESCAN SEM analyzer.

Preparation of N, N' – Bis(Benzoin)- 1,4 butane diimine

To a methanolic solution of 2.12g (0.01mol) benzoin and 1.09g (0.005mol) 1,4 butane diimine was added anhydrous sodium acetate (4g) and the mixture refluxed for an hour. The hot solution was poured into ice-cold water where upon yellow precipitate of the schiff's base separated, it was filtered. Washed with water, dried and recystallised from ethanol[8] .

Preparation of N, N' – Bis(benzoin)- 1,4 butane diimine Metal (II) Complexs

The ethanolic mixture of each(0.01mol) nickel (II) chloride , (0.01mol) zinc (II) chloride and N, N' – bis(benzoin) o-phenylenediimine schiff base (0.01mol) was refluxed separately, followed by drop wise addition of ammonia. The metal chelate separated out was filtered, washed with ethanol followed by ether and dried in an oven at 50°C[8].

Determination of antimicrobial activity

The antimicrobial activity was performed by agar cup plate method.

Microorganisms

Escherichia coli (Gram negative) and fungi as *Candida albicans* were the microorganisms used and they were obtained from the Microbiology Laboratory of the Thanjavur Medical College Hospital, Thanjavur. These microorganisms were identified and confirmed by Microbiologists, Department of Microbiology, Thanjavur Medical College, Thanjavur.

Preparation of 24 hours pure culture

A loop full of each of the microorganisms was suspended in about 10ml of physiological saline in a Roux bottle. Each of these was streaked on to the appropriate culture slants and was incubated at 37ºC for 24 hours except for *Candida albicans* which was incubated at 25ºC for 24-48 hours. After completion of incubation period, when growth was observed the tubes were kept into 2-8°C until use.

Sample solutions for the experiment

Ligand - L

Zinc Metal Complex - Z

Nickel Metal complex -N

The sample solutions as 50µl, 100µl and 150µl were used for the experiment. Standard antibiotic solution as Chloromphenical for bacteria and Nystatin (25mg/ml distilled water- 30µl) for fungi used to compare the test solution.

Preparation of dried filter paper discs

Whattman filter paper (No:1) was used to prepare discs approximately 6 mm in diameter, which are placed in hot air for sterilization After sterilization, the discs were loaded with different concentrations of prepared sample solutions of L, Z and N again kept under refrigeration for 24 hrs.

Application of discs to inoculated agar plates

Previously prepared paper discs were dispensed onto the surface of the inoculated agar plate. Each disc was pressed down firmly to ensure complete contact with the agar surface. The discs were placed on the medium suitably apart and the plates were incubated at 5°C for 1 hr to permit good diffusion and then transferred to incubator at 37°C for 24 hrs. After completion of 24hrs, the plates were inverted and placed in an incubator set to respective temperature for 24 hrs

Antimicrobial assay

Antibiogram was done by disc diffusion method [9,10]. Petri plates were prepared by pouring 30 ml of NA /PDA medium for bacteria/fungi. The test organism was inoculated on solidified agar plate with the help of micropipette and spread and allowed to dry for 10 mints. The surfaces of media were inoculated with bacteria/fungi from a broth culture. A sterile cotton swab is dipped into a standardized bacterial/ fungi test suspension and used to evenly inoculate the entire surface of the Nutrient agar/PDA plate. Briefly, inoculums containing *Escherichia coli* was spread on Nutrient agar plates for bacteria and *Candida albicans* was spread on potato dextrose agar for fungus strains. Using sterile forceps, the sterile filter papers (6 mm diameter) containing the sample (50µl, 100 µl, 150µl and 30µl for respective standard) were laid down on the surface of inoculated agar plate. The plates were incubated at 37◦C for 24 h for the bacteria and at room temperature (30±1) for 24-48 hr. for yeasts strains. Each sample was tested in triplicate.

Measurement of zone of inhibition

The antimicrobial potential of test compounds was determined on the basis of mean diameter of zone of inhibition around the disc in millimeters. The zones of inhibition of the tested microorganisms by the extracts were measured using a millimeter scale. The diameter sizes in mm of the zone of inhibition are shown in the table 2 to 4 and Fig. 2 to 4.

RESULTS AND DISCUSSION

The interaction between metal (II) ions and N, N' – Bis(Benzoin)- 1,4 butane diimine, yielded the desired crystalline, yellow metal (II) complexes compound. These compounds are insoluble in water and common organic solvents, but are readily soluble in acetone.

The IR spectra [Table 1] of the metal (II) complex compounds show bands in the region 1530 - 1570cm^{-1}which are assigned to ν(C==N) stretching vibration, a fundamental feature of azomethine group[11].

The band in the region 1600 - 1660cm^{-1}corresponds to ν(C==C) stretching vibration due to phenyl group, indicating the coordination of bis(benzoin)-1,4 butane diimine Schiff base to the metal (II) ions. The bands within 550 - 590cm^{-1}and 430 - 480cm^{-1}are assigned to ν(M—O) and ν(M—N) stretching vibrations, respectively [12,13] . These bands confirmed the coordination of the Schiff base to the metal (II) ions. The metal (II) complex compounds revealed 1:1 metal to ligand ratio, resulting in a four coordinate complex compounds of which the following molecular structure below is proposed.

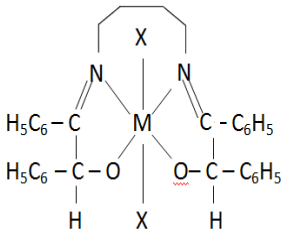

Fig. 1: Shows the molecular structure of bis(benzoin)- 1,4 butane diimine complex compounds

Table 1: The Infrared Spectral Data of the Complex Compounds.

Compound	ν(C-H) (cm^{-1})	ν(C=C) (cm^{-1})	ν(C=N) (cm^{-1})	ν(M-O) (cm^{-1})	ν(M-N) (cm^{-1})
[Ni L']	2922	1616	1568	572	464
[Zn L']	2849	1602	1567	587	449

Table 2: Antimicrobial activity of Ligand

Ligand	*Escherchia Coli* (mm)	*Candida albicans* (mm)
50µl	-	2±0.14
100µl	8± 0.56	4±0.28
150µl	13±0.91	5±0.35
Standard (30µl) (Chloromphenical)	13±0.91	-----
Standard (30µl) (Nystatin)	-----	13±0.91

Table 3: Antimicrobial activity of Zinc complex

Zinc complex	*Escherchia Coli* (mm)	*Candida albicans* (mm)
50µl	-	2±0.14
100µl	2±0.14	4±0.28
150µl	7±0.49	5±0.35
Standard (30µl) (Chloromphenical)	12±0.84	-----
Standard (30µl) (Nystatin)	-----	14±0.98

Table 4: Antimicrobial activity of Nickel complex

Nickel complex	*Escherchia Coli* (mm)	*Candida albicans* (mm)
50µl	-----	-----
100µl	-----	-----
150µl	12±0.84	10±0.7
Standard (30µl) (Chloromphenical)	12±0.84	-----
Standard (30µl) (Nystatin)	-----	14±0.98

Fig. 2: Antimicrobial activity of Ligand

Fig. 3: Antimicrobial activity of Zinc complex

Fig. 4: Antimicrobial activity of Nickel complex

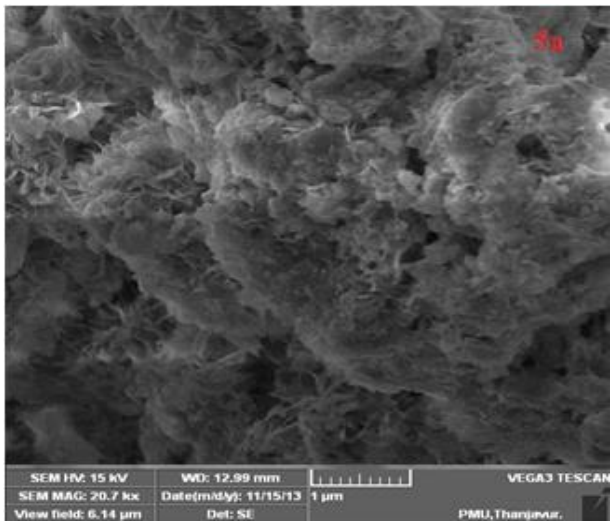

Fig.5a. SEM image of Zinc(II) complex

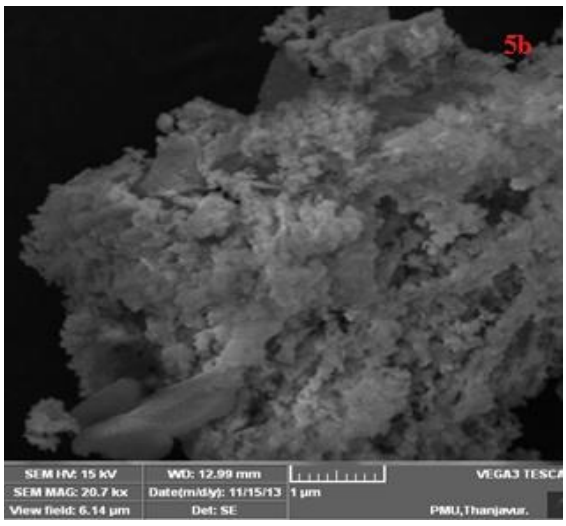

Fig.5b. SEM image of Nickel(II) complex

CONCLUSION

The Schiff base ligand and its Zn(II) and Nickel(II) complexes were prepared. Zn(II) and Nickel(II) complexes were characterized by IR spectra and SEM. The proposed structure of metal complexes has been illustrated in Fig.1. The surface morphology using SEM showed that the particles were polycrystalline with nano sized grains. The antimicrobial activity of ligand and its complexes indicate that both ligand and metal complexes are potentially active against both fungi and bacteria.

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