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Original Research Article

Synthesis, Spectral Characterisation and Antifungal Screening of Mn(II) complex with 4-((2-hydroxynaphthalen-1-ylmethylene) amino)-N-(pyridin-2-yl)benzenesulfonamide

Gomathi Vellaiswamy*, Selvameena Ramaswamy

PG and Research Department of Chemistry, Seethalakshmi Ramaswami College, Tiruchirappalli-620 002, Tamil Nadu, India.

***Corresponding Author:** Gomathi Vellaiswamy, PG and Research Department of Chemistry, Seethalakshmi Ramaswami College, Tiruchirappalli-620 002, Tamil Nadu, India

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ABSTRACT

New Mn(II) complex has been synthesised from the Schiff base 4-((2-hydroxynaphthalen-1-ylmethylene)amino)-N-(pyridin-2-yl)benzenesulfonamide. The composition of the complex has been established by elemental analysis. The ionic nature of the complex has been deduced from conductance measurements. Structural features of the complex has been proposed by magnetic susceptibility measurements, spectral methods (FT-IR, electronic, EI mass) and thermal analysis. The Schiff base behaves as a bidentate N, O donor and coordinated to Mn(II) via the azomethine nitrogen and the phenoxide oxygen. The complex has been screened for antifungal screening towards some clinically important microorganisms.

Keyword: Sulphapyridine; 2-hydroxy-1-naphthaldehyde; Schiff base; Mn(II) complex; antifungal screening

INTRODUCTION

Schiff bases have been extensively used as ligands in coordination chemistry because of their excellent donor abilities [1-4]. Schiff bases are generally excellent chelating agents, [5-10] especially when a functional group like –OH or –SH is present close to the azomethine group so as to form a five or six membered ring with the metal ion. Schiff bases derived from aromatic aldehydes, ortho-substituted with a hydroxyl group have initially aroused the researchers' interest because of their ability to act as bidentate ligands towards transition metal ions [11-14]. Schiff bases and their complexes have a variety of applications in biological, clinical and analytical fields [15-17]. Sulfa drugs are a group of compounds used for eliminating a wide range of infections in human and other animal systems. Sulfur ligands are wide spread among

co-ordination compounds and are important components of biological transition metal complexes which possess many biological applications like diuretic, antiglaucoma and antiepileptic drugs among others [18-20]. Keeping the above facts in mind and in continuation of our research work[21] on transition metal (II) complexes with Schiff bases, in the present paper we report the synthesis and characterisation of Mn(II) complex of Schiff base derived from condensation of 2-hydroxy-1-naphthaldehyde with the well known sulpha drug sulphapyridine. This synthesised complex has been screened for antifungal activities.

MATERIALS AND METHODS

Measurements and Reagents

Melting point was determined using Elico melting point apparatus. Conductivity measurement for the complex was carried out using Elico conductivity bridge and dip type conductivity cell. Magnetic susceptibility was determined using Gouy method-PICO make. IR spectrum of the complex was recorded in KBr pellets with Perkin Elmer IR RXI Spectrometer in the 4000-400 cm^{-1} range. The electronic spectrum was recorded in Perkin Elmer Lambda 35 spectrometer in the 190-1100 nm range. Elemental analysis was performed using elemental analyser. The percentage of metal was determined in ICP Atomic Emission Spectrometer - Thermo Electron IRIS INTREPID II XSP DUO make, with spectral range 165 to >1000 nm. Simultaneous TG and DTA patterns of the complex was recorded on Perkin Elmer STA 6000, Diamond TG/DTA with heating rate 40.00°C to 740.00°C at 10.00°C/min. EI mass spectra were recorded on GC-Mass Spectrometer - Jeol GCMS GC-Mate II make.

Sulphapyridine was purchased from HIMEDIA and 2-hydroxy-1-naphthaldehyde was purchased from Sigma Aldrich-Purity 98.00%. Solvents like DMF and DMSO were purchased from E Merck and used without further purification. Manganese (II) acetate tetrahydrate - $Mn(CH_3COO)_2.4H_2O$ was purchased from Merck. Commercial ethanol was dried over anhydrous quicklime for 24 hours, filtered and distilled before use.

Synthesis

Synthesis of Schiff base 4-((2-hydroxynaphthalen-1-ylmethylene)amino)-N-(pyridin-2-yl)benzenesulfonamide

The synthesis, chemistry and antimicrobial activity of this Schiff base has already been published by our group [21].

Synthesis of $[Mn(L_1\text{-}H)_2]$ complex

The Manganese (II) acetate tetrahydrate (0.613g, 0.0025 mol) dissolved in ethanol was added to the hot stirred solution of ethanol and DMF containing the Schiff base (2.015g, 0.005 mol) in the ratio 1:2(metal:ligand). The mixture was refluxed for 6 hrs on a water bath. The brown precipitate formed during refluxing was cooled in an ice bath and collected by suction filtration, washed thoroughly with ethanol and pet ether and then dried. The resulting complex was mostly insoluble in some common organic solvents and soluble in polar solvents like DMF and DMSO.

Antifungal test by Disc Diffusion Technique

Disc impregnated with known concentration of antibiotic was placed on agar plate that has been inoculated uniformly over the entire plate with culture of the bacterium to be tested. The plate was incubated for 24-48 hrs at 298 K, during this period, the antifungal agent diffuses through the agar and may prevent the growth of the organism. Effectiveness of susceptibility is proportional to the diameter of zone of inhibition. The diameter of the zone of inhibition of growth was measured in mm [22].

RESULTS AND DISCUSSION

Analytical data

The yield and M.Pt for the complex are 60% and 262 °C. The elemental analysis for the complex: Calcd(%); C(61.47), H(3.73), N (9.78), S (7.45),

Mn(6.40) Found(%) C(61.78), H(3.89), N (9.50), S (7.30), Mn(6.35). The elemental analysis results indicate that the metal: ligand stoichiometry is 1:2 for this complex. The molar conductance of the Mn(II) complex is 10.02 ohm^{-1} cm^2 mol^{-1} indicates nonelectrolytic nature of the complex.

Infrared spectral data

The IR spectra of the complex was compared with those of the Schiff base in order to determine the coordination sites that may be involved in chelation. Upon comparison, it is found that the ν (-CH=N) stretching vibration found in the Schiff base at 1618 cm^{-1} is shifted to lower frequency 1598 cm^{-1} indicating the participation of the azomethine nitrogen in coordination. This mode of coordination is further confirmed by non ligand band at 459 cm^{-1} due to ν(M-N) in the complex. The coordination through hydroxyl oxygen after deprotonation is revealed by the disappearence of the broad band at 3428 cm^{-1} and also indicates that there is no lattice/coordinated water molecules in this complex and suggesting four coordinated environment. The coordination of ν(O-H) is further supported by the appearance of non ligand band at 530 cm^{-1} due to ν(M-O) in all the complex [23,24].

Electronic spectral analysis and magnetic susceptibility measurement

The Mn(II) complex (Figure 1) exhibits bands at 265 nm (37735 cm^{-1}), 269 nm (37175 cm^{-1}) and 313 nm (31948 cm^{-1}) due to intraligand transitions and the band at 413 nm (24213 cm^{-1}) due to $^4A_1 \rightarrow {}^4T_1$ transition suggesting four coordinate tetrahedral geometry[25]. The magnetic moment value of 5.62 B.M also supports tetrahedral geometry.

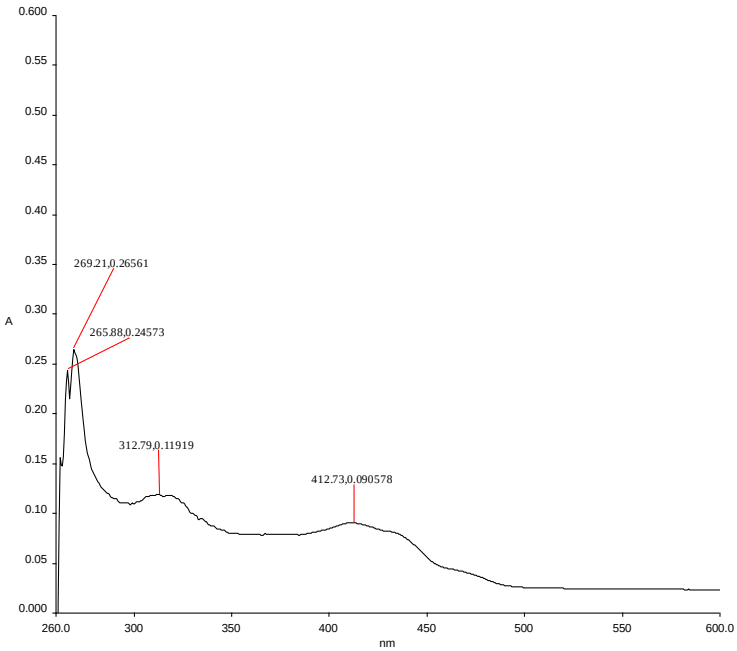

Fig. 1: UV spectrum of complex [Mn(L_1-H)$_2$]

Thermal analysis

Generally in thermogravimetric analysis, lattice water loses at low temperature region between 60-120°C, whereas coordinated water loses in the region 120-250°C. The thermogram of Mn(II) complex has no endothermic peak in the range of 60-250°C. This excludes the possibility of lattice or coordinated water molecules in this complex. The thermogram of Mn(II) complex supports the four coordination around Mn(II) ion and supports tetrahedral geometry.

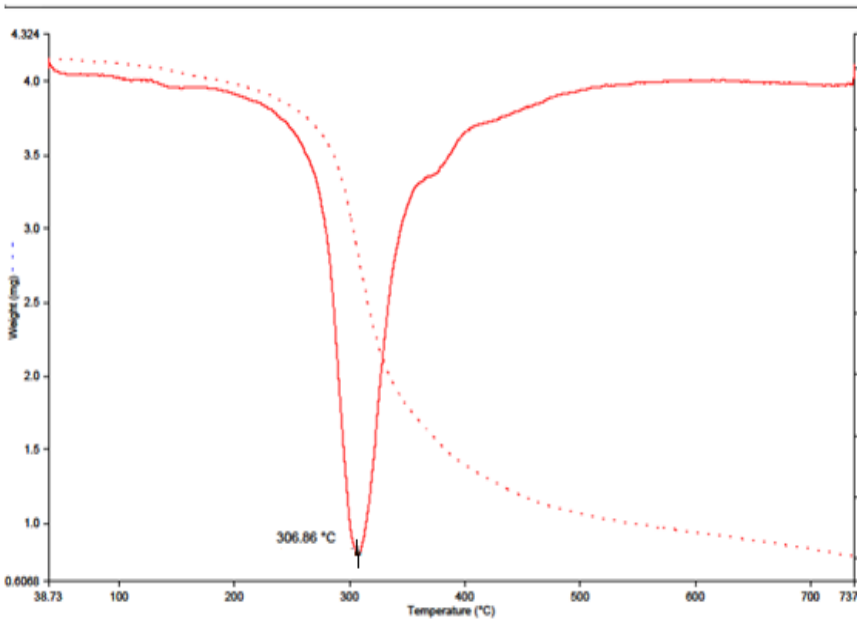

Fig. 2 : Thermogram of complex [Mn(L_1-H)$_2$]

EI Mass spectra

The mass spectra of complex (Figure 3) provide a vital clue for elucidating the formulae of complex. The EI mass spectrum of the metal complex is used to compare their stoichiometry and confirms the probable formulations and the metal: ligand ratio to be 1:2 in the complex. M.Wt. calcd. 858.94 Found. 857.78. The mass spectrum of the complex also confirms the tetrahedral geometry.

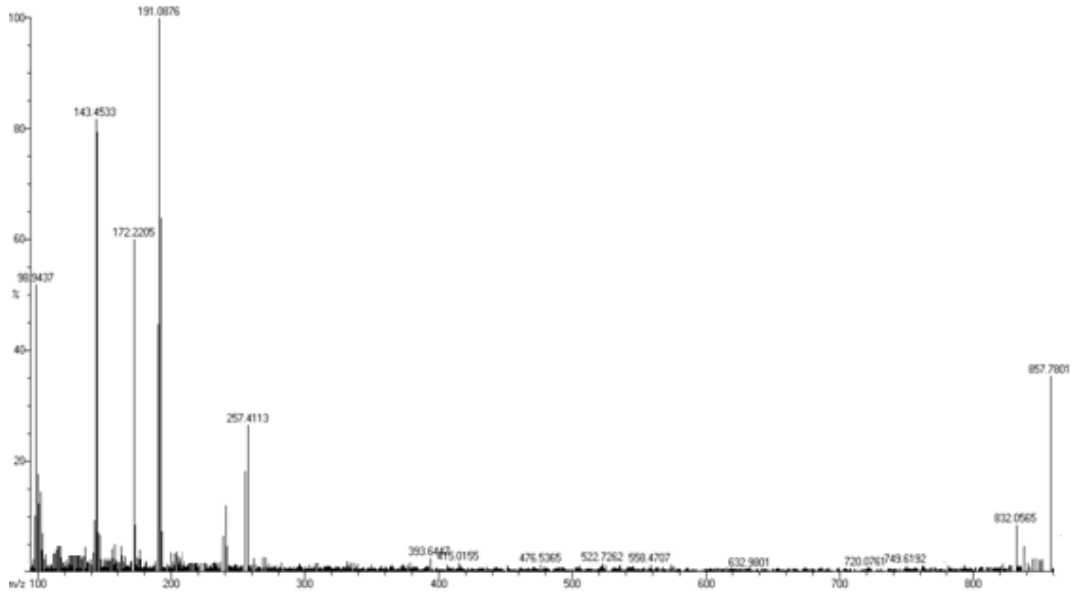

Fig. 3 : EI mass spectrum of complex [Mn(L_1-H)$_2$]

Based on the analytical and spectral evidences the structure has been proposed for the newly synthesized [Mn(L_1-H)$_2$] complex is shown in the Figure 4.

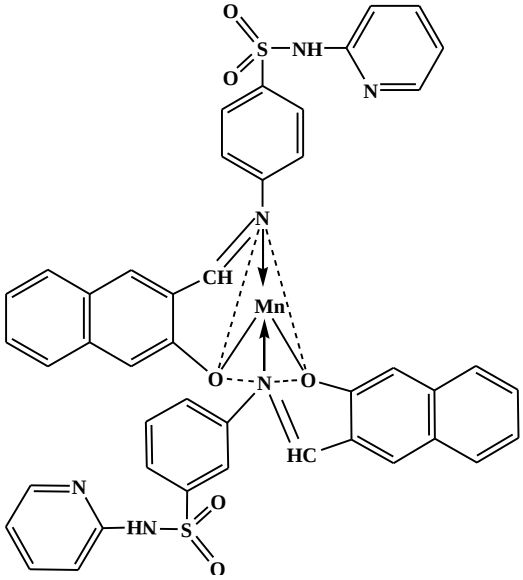

Fig.4: Structure of [Mn(L_1-H)$_2$]

***In-vitro* antifungal bioassay**

The antifungal activity of the [Mn(L_1-H)$_2$] complex was tested against fungal species *Candida albicans and Aspergillus Niger*. The test was carried out in DMSO solution at a concentration of 100 ppm. Nystatin is used as standard at the same concentration. The[Mn(L_1-H)$_2$] complex has been observed to be highly active against *Candida albicans* compared to *Aspergillus Niger*.

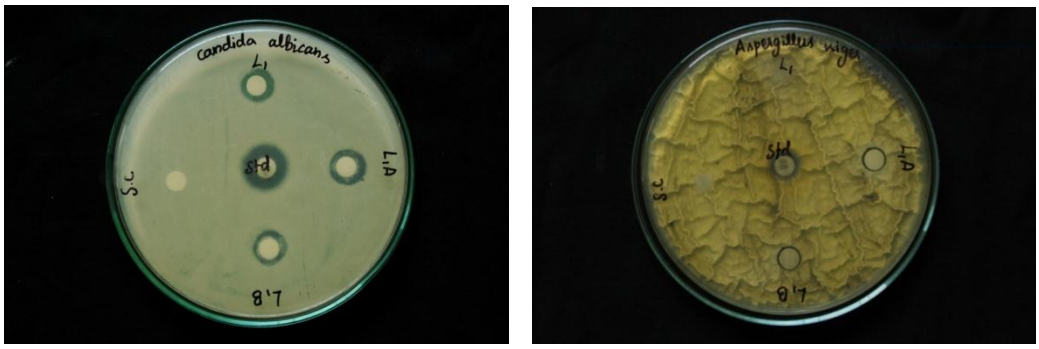

Fig. 5: *In vitro* antifungal screening of [Mn(L_1-H)$_2$] against *Candida albicans and Aspergillus Niger*. [A-Manganese]

Table 1: *In-vitro* Antifungal bioassay of [Mn(L_1-H)$_2$] complex

S.No	Fungal Species	Antifungal activity
1	*Candida albicans*	+++
2	Aspergillus Niger	++
3	*Nystatin*	+++

Highly active = +++ (inhibition zone > 15 mm) Moderatively active = ++ (inhibition zone > 10 mm)
Slightly active = + (inhibition zone > 5 mm)

CONCLUSION

The Mn(II) complex in combination with 4-((2-hydroxynaphthalen-1-ylmethylene)amino)-N-(pyridin-2-yl)benzenesulfonamide has been synthesised and characterised. The Schiff base behaves as a bidentate N, O donor and coordinated to Mn(II) via the azomethine nitrogen and the phenoxide ion. Based on analytical and spectral data, tetrahedral structure has been assigned to the Mn(II)complex. The result of antifungal bioassay indicates that the Mn(II) complex has notable activity against *Candida albicans* compared to *Aspergillus Niger*.

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CONFLICT OF INTEREST STATEMENT

The authors declare that they have no competing interests.

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