

Synthesis, Characterization and Antimicrobial Study of Mannich Base Derived from Piperidine and its Metal Complexes

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ABSTRACT

A ligand derived from piperidine, thiourea and benzaldehyde and its metal complexes with Co(II), Ni(II), Zn(II) and Mn(II) have been synthesized and characterized by IR and thermogravimetry. The antimicrobial activities of the ligand and its metal complexes were determined against the bacteria *Escherichia coli*.

Keywords: Transition metal complex, piperidine, thiourea, benzaldehyde, antimicrobial activity

I. INTRODUCTION

Metal complexes¹ of Mannich bases have been studied extensively in recent years due to the selectivity and sensitivity of ligand towards various biological important metal ions. Mannich reaction is a three component condensation reaction consisting of active hydrogen containing compound.^{2,3} Mannich base complexes have remained an important and popular area of research due to their simple synthesis, adaptability and diverse range of applications. Metal complexes of Mannich bases played a vital role in the development of coordination chemistry. Mannich bases of piperidine exhibit various pharmacological activities⁴ like antimicrobial,⁵ analgesic,⁶ anti-inflammatory,⁷ antiviral,⁸ anticancer,⁹ etc. Organic chelating ligands containing amide moiety as a functional group have a strong ability to form metal

complexes and exhibit a wide range of biological activity.¹⁰

Heterocyclic moieties show a wide range of biological activities. The structural diversity and biological importance of nitrogen-containing heterocyclic moieties have made them attractive synthetic targets.¹¹⁻¹⁵

Thiourea derivatives are selective reagents especially for the determination of transition metal ion complexes. Thiourea has a long history of being used as a ligand in coordination chemistry and to coordinate with a metal via S and N atoms.¹⁶⁻¹⁸

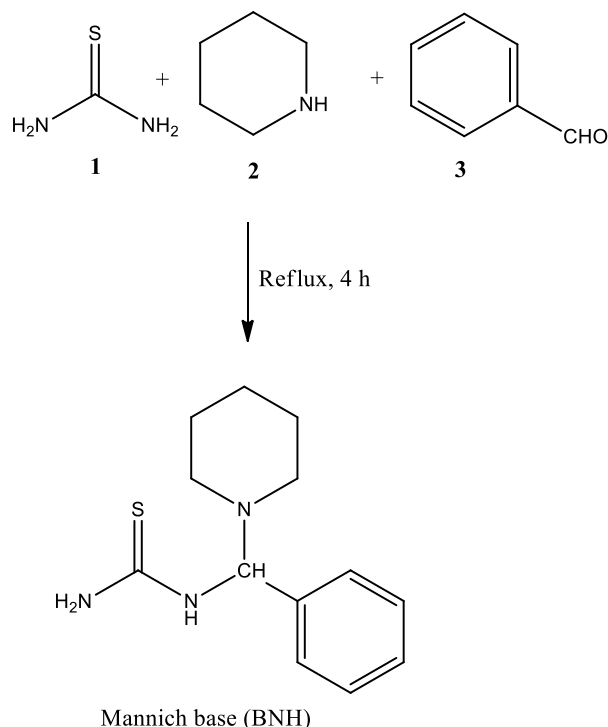
The present paper describes the preparation of Mannich base using thiourea. This work deals with the condensation reaction of thiourea, piperidine and benzaldehyde.¹⁹ The sulphur atom of thiourea along

with the nitrogen of piperidine moiety expected to make it a good ligand for complex formation with transition metals. Thiourea ligands contain the sulphur as well as nitrogen atoms have ability to form the metal complexes.

II. METHODS AND MATERIAL

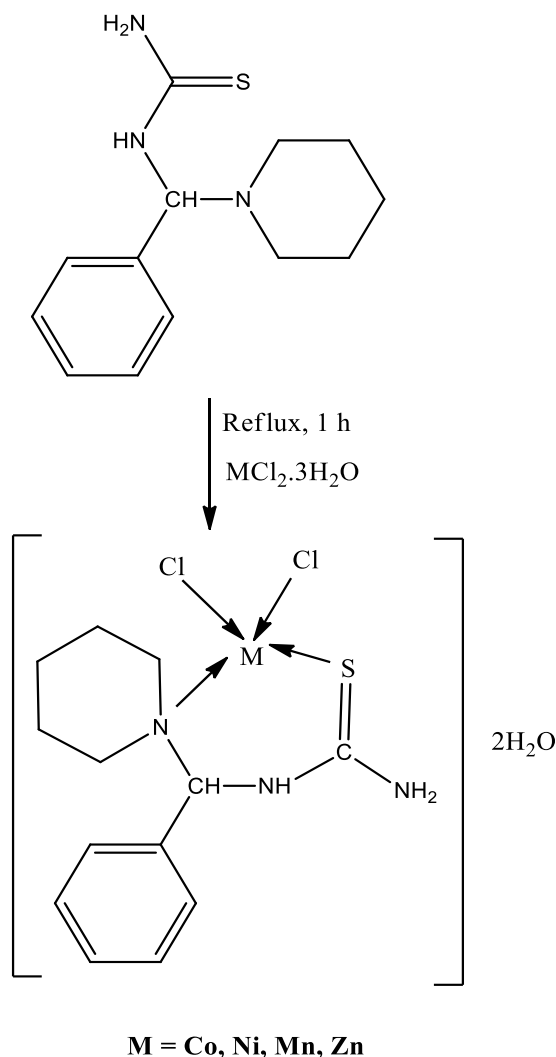
All the chemicals and solvents were of the highest purity and were used as received. IR spectra were recorded with KBr pellets using FT-IR Shimadzu instrument.

Synthesis of Mannich base (BNH): To the methanolic solution of thiourea (1), (0.761 g, 0.01 mol), piperidine (2), (0.852 g, 0.01 mol) was added followed by benzaldehyde (3), (1 mL, 0.01 mol). The reaction mixture was then refluxed with constant stirring at 60 – 70 °C for about 4 h. The colourless solid formed was filtered and washed with methanol. The crude solid obtained was dried and recrystallized using methanol.



Scheme-I: Synthesis of Mannich base (BNH)

Synthesis of metal complexes: To a hot magnetically stirred methanolic solution of Mannich base (0.1 mol), the methanolic solution of metal(II) salt (0.05 mol) was added. The mixture was then refluxed about 1 h. The complex was precipitated, then filtered, washed with methanol then dried and cooled in a desicator.



Scheme-II: Synthesis of Metal complex

III. RESULTS AND DISCUSSION

The IR spectra of the complexes indicates that ligand behaves as a bidentate. In the ligand, the band appearing at 1618 cm^{-1} due to C=S stretching is shifted to lower side at $1557 - 1610\text{ cm}^{-1}$ in the complexes by

confirming the coordination through the sulphur atom of thiourea and not through nitrogen atom.

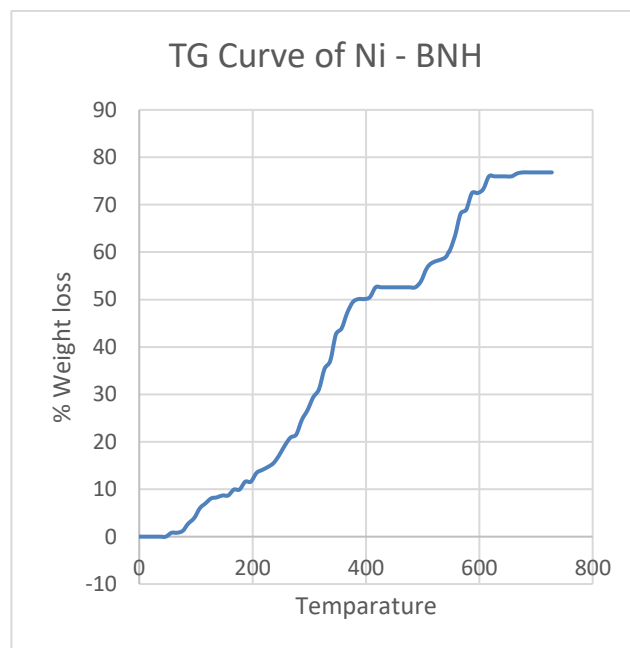
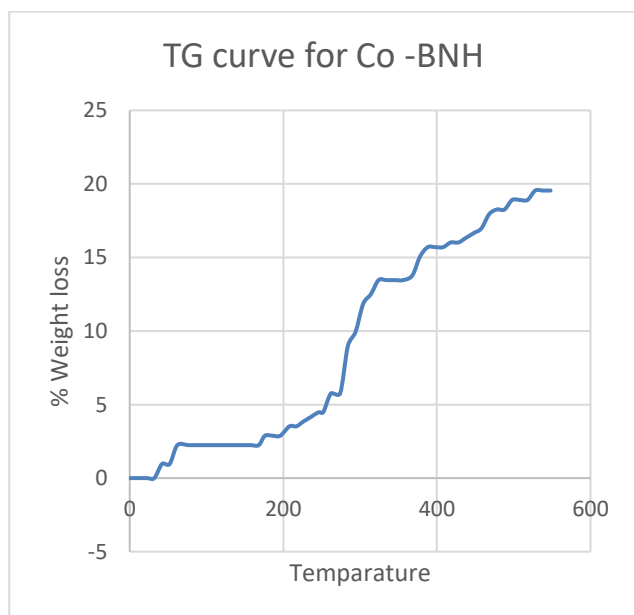
Similarly, in the ligand, the band appearing at 1209-1080 cm^{-1} due to C – N stretching of piperidine is

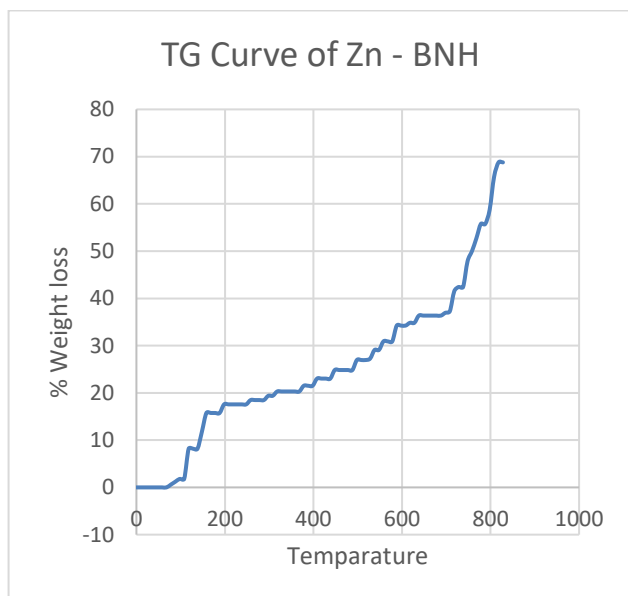
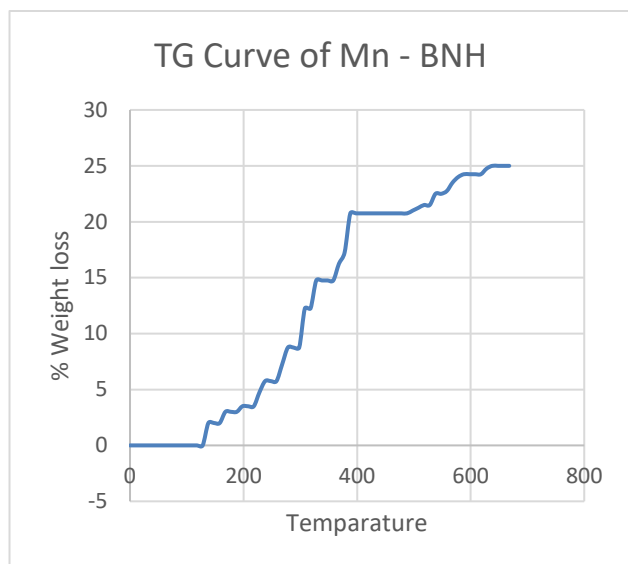
shifted to lower side at 1037 – 1070 cm^{-1} in the complexes by confirming the coordination through the tertiary nitrogen atom of piperidine ring. (Table-1).

Table – 1: IR stretching frequencies of ligand and its metal complexes

Table - 1				
Compound	$\nu(\text{C} = \text{S}) \text{ cm}^{-1}$	$\nu(\text{C-N}) \text{ cm}^{-1}$ of piperidine	$\nu(\text{CH}_2) \text{ cm}^{-1}$ of piperidine	$\nu(\text{M} - \text{N}) \text{ cm}^{-1}$
BNH (Ligand)	1618	1209-1080	2945	--
Co- BNH	1557	1070	2866	445
Ni- BNH	1602	1043	2820	524
Mn- BNH	1564	1053	2831	515
Zn- BNH	1610	1037	2850	455

Thermogravimetric Analysis (TGA): All the metal complexes were analyzed by dynamic thermogravimetry. In TGA we measure weight loss with respect to temperature. The TG curve suggested that weight loss corresponds for water molecules and temperature range indicates that the nature of water molecule is lattice.





Antibacterial activity: All the compounds were screened in vitro for their antimicrobial activity against *Escherichia coli* by well diffusion method. The zone of inhibition values were found out at the end of 24 h at 37 °C for bacterial stains. The antibacterial data (Table- 2) (Fig. 1) suggested that Mannich base of piperidine and its complexes with zinc and cobalt metal were found to be biologically active. It is observed that activity of metal complexes of Mannich bases are good when compared with the ligand.

Table – 2 Antibacterial activity of ligand and its metal complexes

Name	Compound	Zone of inhibition	Anti bacterial activity
Standard	Streptomycin	12 mm	Active
C	Zn- BNH	5 mm	Active
A	Mn- BNH	0 mm	Inactive
D	Ni- BNH	0 mm	Inactive
4	Co- BNH	1.5 mm	Active
5	BNH (Ligand)	1 mm	Active



Figure 1: Zone of inhibition of antibacterial activity

IV. CONCLUSION

The ligand BNH and its metal complexes have been synthesized and characterized by IR and Thermogravimetry techniques. It is indicated from IR studies that the ligand is coordinated to metal as a bidentate ligand. The TG curve indicates the presence of two water molecules which are lattice water. The antibacterial screening of ligand shows inhibition in the growth of *E. Coli*. The metal complexes show greater activity than the ligand.

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