



# Physico-chemical and antimicrobial study of metal complexes of Ni (II) and Schiff bases derived from heterocyclic amines.

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## ABSTRACT

The Schiff bases like benzaldimine pyrrole was synthesized by benzaldehyde and pyrrole. benzaldimine thiophene was synthesized by benzaldehyde and thiophene. benzaldimine furan was synthesized by benzaldehyde and furan. benzaldimine pyridine was synthesized by benzaldehyde and pyridine. Naphthaldimine pyrrole was synthesized by naphthaldehyde and pyrrole. Naphthaldimine thiophene was synthesized by naphthaldehyde and thiophene. Naphthaldimine furan was synthesized by naphthaldehyde and furan. Naphthaldimine pyridine was synthesized by naphthaldehyde and pyridine. Nickel complexes of all bases were made and antimicrobial activity screening have been studied.

Key Word: Antimicrobial, Schiff base, nickel, heterocyclic amine, complex

## INTRODUCTION

Co-ordination compounds play a very significant role in our lives. The quest for deeper understanding of the nature of chemical bond has provided a major and contributing incentive in the chemical sciences particularly in the study of co-ordination compounds.

The chemistry of metal complexes with multidentate ligands having delocalized d orbital, such as Schiff bases has recently gained more attention because of their numerous uses. (Lu and Yang 2003, Wu and co-worker 2002)

Transitional metals play a vital role in different biological processes. Some of these processes are very specific in requirements of metal ions. Only certain metal ions in the specific oxidation states can accomplish the necessary catalytic structure requirement. The activity of various metal ions in biological system has been explained in term of complex species formed in living organism. Many metal ions are found in several enzymes and reported to play a major role in various enzymatic reaction. (Chandra & Sharma 2007) The metal complexes formed by ligand containing Sulphur, nitrogen and oxygen are immense importance due to exhibiting anticancer (Chaudhri & Sahani 1976), antiviral (Frend & Blenj 1966), antibacterial (Kumari & Prakash 2011, Verma & coworker20) and antifungal (Ferrow and co-worker 1954)

The Schiff bases derived from some sulpha drugs have been successfully used as chelating, bacterial and fungicidal agents (Goyal & Lal 1989). Dash et al (1985) reported the antifungal activity of heterocyclic Schiff bases derived from 4,5 diaryl-2-amino thiozole and substituted aldehyde. Agarwal and co-worker (1981) synthesized some coumarines and hydrazones as antibacterial and antifungal agents against *B. subtilis* and *I. montagrophytes*. Fungicidal activity (Giri and co-worker 1981) of N-aryl or hetero-2,2-disubstituted azomethine was tested against *A. niger* and *A. flavus*. Ibrahim and co-worker (1994) investigated the antimicrobial and antifungal activities.

## MATERIAL AND METHOD

### A. Synthesis of Schiff Bases:

#### I. Synthesis of Benzaldimine Pyrrole

Benzaldehyde (0.01 mol) and ortho amino pyrrole (0.01 mol) were mixed in 50 ml of ethyl alcohol. The mixture was refluxed for 3-7 hours on a water bath. The resulting content was filtered to get white coloured crystal which were washed with ether and then recrystallized from ethanol. The crystals were then dried under reduced pressure over fused calc. chloride.

#### II. Synthesis of Benzaldimine Thiophene

Benzaldehyde (0.01 mol) and ortho amino thiophene (0.01 mol) were mixed in 50 ml of ethyl alcohol. The mixture was refluxed for 3-7 hours on a water bath. The resulting content was filtered to get white coloured crystal which were washed with ether and then recrystallized from ethanol. The crystals were then dried under reduced pressure over fused calc. chloride.

#### III. Synthesis of Benzaldimine Furan

Benzaldehyde (0.01 mol) and ortho amino furan (0.01 mol) were mixed in 50 ml of ethyl alcohol. The mixture was refluxed for 3-7 hours on a water bath. The resulting content was filtered to get white coloured crystal which were washed with ether and then recrystallized from ethanol. The crystals were then dried under reduced pressure over fused calc. chloride.

#### IV. Synthesis of Benzaldimine Pyridine

Benzaldehyde (0.01 mol) and ortho amino pyridine (0.01 mol) were mixed in 50 ml of ethyl alcohol. The mixture was refluxed for 3-7 hours on a water bath. The resulting content was filtered to get white coloured crystal which were washed with ether and then recrystallized from ethanol. The crystals were then dried under reduced pressure over fused calc. chloride.

### V. Synthesis of Naphthaldimine Pyrrole

Naphthaldehyde (0.01 mol) and ortho amino pyrrole (0.01 mol) were mixed in 50 ml of ethyl alcohol. The mixture was refluxed for 6-7 hours on a water bath. The resulting content was filtered to get yellow coloured crystal which were washed with ether and then recrystallized from ethanol. The crystals were then dried under reduced pressure over fused calc. chloride.

### VI. Synthesis of Naphthaldimine Thiophene

Naphthaldehyde (0.01 mol) and ortho amino thiophene (0.01 mol) were mixed in 50 ml of ethyl alcohol. The mixture was refluxed for 6-7 hours on a water bath. The resulting content was filtered to get yellow coloured crystal which were washed with ether and then recrystallized from ethanol. The crystals were then dried under reduced pressure over fused calc. chloride.

### VII. Synthesis of Naphthaldimine Furan

Naphthaldehyde (0.01 mol) and ortho amino furan (0.01 mol) were mixed in 50 ml of ethyl alcohol. The mixture was refluxed for 6-7 hours on a water bath. The resulting content was filtered to get yellow coloured crystal which were washed with ether and then recrystallized from ethanol. The crystals were then dried under reduced pressure over fused calc. chloride.

## VIII. Synthesis of Naphthaldimine Pyridine

Naphthaldehyde (0.01 mol) and ortho amino pyridine (0.01 mol) were mixed in 50 ml of ethyl alcohol. The mixture was refluxed for 6-7 hours on a water bath. The resulting content was filtered to get yellow coloured crystal which were washed with ether and then recrystallized from ethanol. The crystals were then dried under reduced pressure over fused calc. chloride.

### (B) Synthesis of Ni (II) complexes

#### I. Synthesis of Benzaldimine Pyrrole Ni (II) complex

Benzaldehyde (0.01 mol) dissolved in 20 ml of ethanol, benzaldimine pyrrole (0.01 mol) in 20 ml of ethanol and NiCl<sub>2</sub> (0.01 mol) in 10 ml of water and ethanol were taken together in flask. The content was refluxed for 8-10 hours on a water bath and solution was reduced to 1/3 volume. Greenish coloured crystalline solid so obtained was filtered, washed with ether and recrystallized with ethanol and dried in desiccator over fused CaCl<sub>2</sub>.

#### II. Synthesis of Benzaldimine Thiophene Ni (II) complex

Benzaldehyde (0.01 mol) dissolved in 20 ml of ethanol, benzaldimine thiophene (0.01 mol) in 20 ml of ethanol and NiCl<sub>2</sub> (0.01 mol) in 10 ml of water and ethanol were taken together in flask. The content was refluxed for 8-10 hours on a water bath and solution was reduced to 1/3 volume. Greenish coloured crystalline solid so obtained was filtered, washed with ether and recrystallized with ethanol and dried in desiccator over fused CaCl<sub>2</sub>.

#### III. Synthesis of Benzaldimine Furan Ni (II) complex

Benzaldehyde (0.01 mol) dissolved in 20 ml of ethanol, benzaldimine furan (0.01 mol) in 20 ml of ethanol and NiCl<sub>2</sub> (0.01 mol) in 10 ml of water and ethanol were taken together in flask. The content was refluxed for 8-10 hours on a water bath and solution was reduced to 1/3 volume. Greenish coloured crystalline solid so obtained was filtered, washed with ether and recrystallized with ethanol and dried in desiccator over fused CaCl<sub>2</sub>.

#### IV. Synthesis of Benzaldimine Pyridine Ni (II) complex

Benzaldehyde (0.01 mol) dissolved in 20 ml of ethanol, benzaldimine pyridine (0.01 mol) in 20 ml of ethanol and NiCl<sub>2</sub> (0.01 mol) in 10 ml of water and ethanol were taken together in flask. The content was refluxed for 8-10 hours on a water bath and solution was reduced to 1/3 volume. Greenish coloured crystalline solid so obtained was filtered, washed with ether and recrystallized with ethanol and dried in desiccator over fused CaCl<sub>2</sub>.

#### V. Synthesis of Naphthaldimine Pyrrole Ni (II) complex

Naphthaldehyde (0.01 mol) dissolved in 20 ml of ethanol, naphthaldimine pyrrole (0.01 mol) in 20 ml of ethanol and NiCl<sub>2</sub> (0.01 mol) in 10 ml of water and ethanol were taken together in flask. The content was refluxed for 8-10 hours on a water bath and solution was reduced to 1/3 volume. Greenish coloured crystalline solid so obtained was filtered, washed with ether and recrystallized with ethanol and dried in desiccator over fused CaCl<sub>2</sub>.

#### VI. Synthesis of Naphthaldimine Thiophene Ni (II) complex

Naphthaldehyde (0.01 mol) dissolved in 20 ml of ethanol, naphthaldimine thiophene (0.01 mol) in 20 ml of ethanol and NiCl<sub>2</sub> (0.01 mol) in 10 ml of water and ethanol were taken together in flask. The content was refluxed for 8-10 hours on a water bath and solution was reduced to 1/3 volume. Greenish coloured crystalline solid so obtained was filtered, washed with ether and recrystallized with ethanol and dried in desiccator over fused CaCl<sub>2</sub>.

#### VII. Synthesis of Naphthaldimine Furan Ni (II) complex

Naphthaldehyde (0.01 mol) dissolved in 20 ml of ethanol, naphthaldimine furan (0.01 mol) in 20 ml of ethanol and NiCl<sub>2</sub> (0.01 mol) in 10 ml of water and ethanol were taken together in flask. The content was refluxed for 8-10 hours on a water bath and solution was reduced to 1/3 volume. Greenish coloured crystalline solid so obtained was filtered, washed with ether and recrystallized with ethanol and dried in desiccator over fused CaCl<sub>2</sub>.

#### IV. Synthesis of Naphthaldimine Pyridine Ni (II) complex

Naphthaldehyde (0.01 mol) dissolved in 20 ml of ethanol, naphthaldimine pyridine (0.01 mol) in 20 ml of ethanol and  $\text{NiCl}_2$  (0.01 mol) in 10 ml of water and ethanol were taken together in flask. The content was refluxed for 8-10 hours on a water bath and solution was reduced to 1/3 volume. Greenish coloured crystalline solid so obtained was filtered, washed with ether and recrystallized with ethanol and dried in desicator over fused  $\text{CaCl}_2$ .

#### METHOD

The method includes addition of test compound in variable concentration in separate lots of sterilized nutrients medium and thereafter the medium containing chemical is plated with micro-organism. The result is taken after a period of growth at  $28 \pm 10^\circ\text{C}$  temperature for the appearance of growth.

#### MEDIUM

For the evaluation of antimicrobial activity potato dextrose agar (PDA) and nutrient agar (NA) were prepared. The prepared medium was sterilized at  $15 \text{ lb}$ ,  $121^\circ\text{C}$  temperature for 30 minutes in autoclave.

Before pouring the medium in sterilized petridishes 0.001 gm of sterilized medium in each petridish. The medium solidified after 30 minutes.

#### INCUBATION

The prepared petridishes were incubated at  $38 \pm 10^\circ\text{C}$  for fungi and  $37 \pm 10^\circ\text{C}$  for bacteria in BOD incubator in separate lots.

#### Preparation of Sample Solution

Calculated amount of Schiff bases and their metal complexes were dissolved in suitable volume of methanol. So as to make two different concentrations from 500-1000 ppm.

#### RESULT AND DISCUSSION

All the results of antibacterial and antifungal screening have been summarized in Table 1 to 8. The selected pathogens are as 1. *A. niger* 2. *A. flavus* 3. *S. aureus* 4. *E. coli* 5. *B. subtilis*

Table-1: Antifungal and antibacterial activity screening of ligands and their complexes.

S.N.	Name of ligands/complex	Concentration in ppm	Antifungal		Antibacterial		
			<i>A. lavus</i>	<i>A. niger</i>	<i>E. coli</i>	<i>S. aureus</i>	<i>B. subtilis</i>
1	Benzaldimine pyrrole	250	-	-	-	-	-
		500	+	-	-	-	++
		750	++	++	-	+++	++
		1000	+++	+++	-	++++	++++
2	Nickel complex of Benzaldimine pyrrole	250	+	+	-	+	+
		500	++	++	-	++	++
		750	++	++	-	++	++
		1000	++++	++++	-	++++	++++

Table-2: Antifungal and antibacterial activity screening of ligands and their complexes

S.N.	Name of ligands/complex	Concentration in ppm	Antifungal		Antibacterial		
			A. lavus	A. niger	E. coli	S. aureus	B. subtilis
1	Benzaldimine furan	250	-	-	-	-	-
		500	+	-	-	-	++
		750	++	++	-	+++	++
		1000	+++	+++	-	++++	+++
2	Nickel complex of Benzaldimine furan	250	+	+	-	+	+
		500	++	++	-	++	++
		750	++	++	-	++	++
		1000	++++	++++	-	++++	++++

Table-3 Antifungal and antibacterial activity screening of ligands and their complexes: =

S.N.	Name of ligands/complex	Concentration in ppm	Antifungal		Antibacterial		
			A. lavus	A. niger	E. coli	S. aureus	B. subtilis
1	Benzaldimine thiophene	250	-	-	-	-	-
		500	+	-	-	-	++
		750	++	++	-	+++	++
		1000	+++	+++	-	++++	++++
2	Nickel complex of Benzaldimine thiophene	250	+	+	-	+	+
		500	++	++	-	++	++
		750	++	++	-	++	++
		1000	++++	++++	-	++++	++++

Table-4: Antifungal and antibacterial activity screening of ligands and their complexes

S.N.	Name of ligands/complex	Concentration in ppm	Antifungal		Antibacterial		
			A. lavus	A. niger	E. coli	S. aureus	B. subtilis
1	Benzaldimine pyridine	250	-	-	-	-	-
		500	+	-	-	-	++
		750	++	++	-	+++	++
		1000	+++	+++	-	++++	++++
2	Nickel complex of Benzaldimine pyridine	250	+	+	-	+	+
		500	++	++	-	++	++
		750	++	++	-	++	++
		1000	++++	++++	-	++++	++++

Table-5: Antifungal and antibacterial activity screening of ligands and their complexes

S.N.	Name of ligands/complex	Concentration in ppm	Antifungal		Antibacterial		
			A. lavus	A. niger	E. coli	S. aureus	B. subtilis
	Naphthaldimine pyrrole	250	-	-	-	-	-
		500	+	-	-	-	++
		750	++	++	-	+++	++
		1000	+++	+++	-	++++	++++
	Nickel complex of Naphthaldimine pyrrole	250	+	+	-	+	+
		500	++	++	-	++	++
		750	++	++	-	++	++
		1000	++++	++++	-	++++	++++

Table-6: Antifungal and antibacterial activity screening of ligands and their complexes

S.N.	Name of ligands/complex	Concentration in ppm	Antifungal		Antibacterial		
			A. lavus	A. niger	E. coli	S. aureus	B. subtilis
1	Naphthaldimine furan	250	-	-	-	-	-
		500	+	-	-	-	++
		750	++	++	-	+++	++
		1000	+++	+++	-	++++	++++
2	Nickel complex of Naphthaldimine furan	250	+	+	-	+	+
		500	++	++	-	++	++
		750	+++	+++	-	++	++
		1000	++++	++++	-	++++	++++

Table-7: Antifungal and antibacterial activity screening of ligands and their complexes

S.N.	Name of ligands/complex	Concentration in ppm	Antifungal		Antibacterial		
			A. lavus	A. niger	E. coli	S. aureus	B. subtilis
1	Naphthaldimine thiophene	250	-	-	-	-	-
		500	+	-	-	-	++
		750	++	++	-	+++	++
		1000	+++	+++	-	++++	++++
2	Nickel complex of Naphthaldimine thiophene	250	+	+	-	+	+
		500	++	++	-	++	++
		750	++	++	-	++	++
		1000	++++	++++	-	++++	++++

Table-8: Antifungal and antibacterial activity screening of ligands and their complexes

S.N.	Name of ligands/complex	Concentration in ppm	Antifungal		Antibacterial		
			A. lavus	A. niger	E. coli	S. aureus	B. subtilis
1	Naphthaldimine pyridine	250	-	-	-	-	-
		500	++	-	-	-	++
		750	++	++	-	+++	++
		1000	+++	+++	-	++++	++++
2	Nickel complex of Naphthaldimine pyridine	250	+	+	-	+	+
		500	++	++	-	++	++
		750	++	++	-	++	++
		1000	++++	++++	-	++++	++++

Among the synthesized Schiff bases and their metal complexes all of them exhibited better activity against the selected pathogenic organism. The detailed antimicrobial study of these synthesized Schiff bases revealed that all these are active against fungi and bacteria. Metal complexes of Nickel exhibited enhanced activity.

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