# Chemical Synthesis and Microbial Evaluation of Iron (II) and Cobalt (II) Complexes of Schiff Base Dervived from B-Hydroxynapthalene-1-Carbaldehyde Using 2-Aminoaniline and 2-Hydroxyaniline as the Aniline Di-Substituents

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Abstract: Chemical synthesis and microbial evaluation of Iron (II) and Cobalt (II) complexes of Schiff base derived from  $\beta$ -hydroxynapthalene-1-carbaldehyde was carried out using 2-aminoaniline and 2-hydroxylaniline as the substituted aniline. The chemicals used for the synthesis and complexation were of analytical grade and were used as supplied without further purification. The infrared (IR), Ultraviolet Visible (UV-Vis) and Mass spectroscopic (MS) analysis were carried out on the half unit ligands (½LOA, ½LNA), Schiff bases (LOA, LNA) and their respective complexes (FeOcplx, FeNcplx for Iron (II) and CoOcplx, CoNcplx for Cobalt (II)). The UV-Vis results show double peaks in all the complexes except FeOcplx and ligands with singlets and LNA with triplet attributed to  $n \rightarrow \pi^*$  and  $\pi \rightarrow \pi^*$  transition. The IR absorption show characteristic behavior in the sense that, the OH group found in both the half unit ligands and Schiff base within the region (3262.959-3330.483) cm<sup>-1</sup> were absent in the complexes due to the usage of carbonyl oxygen in complexation. Likewise the v (N-H) of primary amine detected in half unit ligands: ½LNA (3414.764cm<sup>-1</sup>) and ½LOA (3495.558cm<sup>-1</sup>) were also absent due to bonding process. The MS of the compounds show greater antimicrobial activity on both the fungi and bacteria than the ligands.

Keywords: β-hydroxynapthalene-1-carbaldehyde, 2-aminoaniline, 2-hydroxylaniline, Complexation

## 1. Introduction

Chelation chemistry has wide range of application in biochemical systems, solvent extractions, ion exchange, precipitation etc [1], and involves the formation or presence of two or more separate coordinate bonds between a polydentate ligands and a single central metal atom. One of the most recently cited coordination compound in chelation is Schiff base which serve as synthetic intermediates and as ligands for transition and inner transition metal ions [2]. The Schiff base may function as a bidentate, N, O<sup>-†</sup> tridentate N, O, O<sup>-†</sup> N, O, N <sup>-†</sup> N, O, S <sup>-†</sup> tetradentate N, N, O, O<sup>-†</sup> and hexadentate N, N, O, O, S, S <sup>-†</sup> donor ligands [3, 4] and characterized by the presence of a C=N double bond (imine) to an aromatic group through C or N atom to avoid rapid decomposition or polymerization [5]

Metal Schiff base complexes obtained from Bhydroxynaphthalane-1-carbaldehyde has been revealed to show broad areas of biological effectiveness, catalytic reaction, sensors, electrode and fluorescence [8]. The metal complexes possess good biological activities like antimicrobial, anti-inflammatory, analgesic, diuretic and anti-viral activities [9].

Di-substituted anilines: 2-hydroxyanilines and 2aminoanilines are organic bases with nucleophilic atoms (O, N) attached to phenyl group respectively. They exhibit good quality of coordination and are capable of forming stable metallic complexes [6-9] when synthesized with 2hydroxynopthalene-1-carbaldehyde.

This research work focuses on synthesis, characterization and anti microbial study of Fe (II) Co (II) complexes of Schiff base derived from 2-hychoxynaphthalene-1carbaldehyde and two di-substituted anilines: 2aminoaniline and 2-hydroxyl aniline.

## 2. Experimental

## 2.1 The Apparatus and Reagents

The reagents used for this research were of analytical standard. They include 2-hydroxynapthalene-1carbaldehyde (Sigma-Aldrich), 2-aminoaniline (J. T. Baker), 2-hydroxyaniline (Sigma Aldrich), and salicylaldehyde (J. T. Baker). Others were Cobalt (II) Acetate tetrahydrate (British Drug House, BDH), Iron (II) acetate tetrahydrate (BDH), Piperidine (J. T. Baker), and DMSO (S. Aldrich). The solvent were ethanol (J. T. Baker), Methanol (J. T. Baker), Acetone (J. T. baker) and Ether (J. T. Baker).

The electronic equipments: Golden mettle USA Electronic balance model 3002, Magnetic Stirrer Hot Plate 79-1 Techmel and Techmel USA, J. J. I Precision Force Electronic mixer (china), Melting point apparatus, HACH

HO4Od conductivity meter, IR-Fourier Transformed Infrared Spectrophotometer, LE-440 Elemental Analzer (Exeter Analytical Inc. USA), Agilent 6840A gas chromatogragh, 5973C inert mass spectrometer (Agilent technologies) Incubator E-track scientific instrument, England; Autoclave, E-track scientific instrument England; Microscope OPTIKA Italy, SDA-livesaver (lis) Biotech SanDiego CA 92121, USA; Mueller Hinton agar-Titan Biotech Itd, Rajasthem, India; Nutrient agar-Tm media, Titan Biotech Ltd Rajasthem India.

## The bacteria Species:

Salmonella enterica (gram negative) Escherichia coli (gram negative), Staphylococcus aureus (gram positive) and Bacillus subtilis (gram positive) The fungal species: Aspergilus flavus, Fusarium monoliforme, Rhizopus stolonifer and Fusarium solani.

## 2.2 Preparation of ligands;

The preparation of the ligands for complexation was done stewisely.

Preparation of half unit ligands of 2-aminoaniline and 2hydroxyaniline (½LNA, ½ LOA) fig 1 (a)



Fig 1 (a) Half ligand of 2-aminoaniine and 2hydroxyaniline (b) Schiff base ligands of 2-aminoaniline and 2-hydroxyaniline

A solution of 20 mmole each of the two anilines (2.16g 2aminoaniline or 2.18 g of 2-hydroxyaniline) were obtained separately with 150ml of ethanol. These were added slowly with stirring.6.89g (20 mmole) of 2hydroxynapthalene-1-carbaldehyde was dissolved in 100ml anhydrous ethanol and the two solution were mixed and stirred for 30 minutes and then reflux for 30 minutes. The solid was filtered, washed and recrystallised with mixed solvent (1:1:1) ratio of methanol, ethanol and acetone. The percentage yield and melting point were determined [6, 10].

## (b) Preparation of unsymmetrical Schiff base ligands (LNA, LOA) fig 1 (b) [6, 10, 11].

A solution of 10 mmole of the half ligands ( $\frac{1}{2}$  LNA 2.62g or  $\frac{1}{2}$ LOA 2.59g) was prepared in anhydrous ethanol and a solution of 10 mmole (1.22g) of salicylaldehyde was also prepared in anhydrous ethanol. The two solution were mixed and refluxed for 1 hour. The resultant product was

concentrated in vacuum until it precipitated a coloured solid crystals. It was further recrystallized to increase its purity. Their colors, melting point and percentage yield were determined.

#### (c) Complexation

A 1 mmole solution of the metal acetate, M (OAc)  $_{2:}$  (0.217g Co (OAc)  $_{2}$  or (0.216g Fe (OAc)  $_{2}$  was placed in boiling ethanol solvent 1 mmole solution of the unsymmetrical Schiff base ligands: (LNA 0.416g or LOA 0.419g) was added. Then a few drops of piperidine were also added and refluxed the mixture for 1 hour. At the end of refluxing, the mixture was allowed to stand in other to allow the colored crystals to precipitate, then wash with cold methanol and cool ether and dry in an oven at  $50^{\circ}$ c.

#### 2.3 Instrumental analysis

The molar conductivity measurements of the samples were carved out with a  $10^{-3}$ M solution of the samples in absolute ethanol and in acetone at  $25 + 0.5^{\circ}$ C. The conductivity was determined using a conductivity meter: HACH HQ 40d. The UV-VIS spectra of the complexes

Volume 10 Issue 8, August 2021 <u>www.ijsr.net</u> Licensed Under Creative Commons Attribution CC BY  $(10^{-3}M)$  in absolute ethanol were recorded on a spectrophotometer: Spectrum lab 725s UV-VIS spectrophotometer, and scanned in the region between 200-880nm at a spectral band width of 2nm; and the absorbance versus wavelength data of the plot were recorded.

The FT-IR Spectra of the samples were obtained in the region of 4000-650 cm<sup>-1</sup> range using cary 630FT-IR Spectrometer, equipped with KBr optics and complimentary diamond ATR accessories. The HappGenzel function was used for apodization. Agilant Microlab Expert FTIR spectrometer software was used to acquire and process the data.

The elemental contents, C, H, N, O, of the samples were determined by dynamic flash combustion, using an elemental analyzer: LE-440 Elemental Analyzer, Exeter Analytical Inc. UK. The combustion and reduction temperature were 975 °C and 600 °C respectively while oven temperature was 81°C. The chromatographic column was Porapak PQS column, while the detector was thermal conductivity detector. The G. C. mass spectrometry of the samples were done using Agilent 6890A gas chromatograph, coupled to 5973 C inert mass spectrometer with triple axis detector and electron impact source. All the instrumental analysis were carried out in ISI Analytical laboratory Lagos, Nigeria [12]

## Reaction with Cobalt to form Cobalt (II) Complex of Schiff base

(a) Formation of Co complex with hydroxylaniline







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#### (b) Formation of Fe Complex with 2-aminophenol



Diameter	of	Inbition	Zone	(mm)
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S/No	Compound	Aspergillus flavus		Fusarium moniliforme			Rhizopus stolonifer			Fusarium solani			
		Concent	ration (m	ig/ml)	Concen	tration (m	g/ml)	Concer	ntration (n	ig/ml)	Concentration (mg/ml)		
		50	25	12.5	50	25	12.5	50	25	12.5	50	25	12.5
1	½LNA	7	7	-ve	7	-ve	-ve	-ve	-ve	-ve	7	-ve	-ve
2	½LOA	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
3	LNA	7	-ve	-ve	7	-ve	-ve	-ve	-ve	-ve	7	-ve	-ve
4	LOA	-ve	-ve	-ve	8	-ve	-ve	-ve	-ve	-ve	7	-ve	-ve
5	FeOCplx	8	7	7	10	8	8	9	9	-ve	10	9	-ve
6	FeNCplx	10	9	-ve	11	9	7	11	10	8	11	10	-ve
7	CoOCplx	10	8	-ve	12	9	-ve	8	7	-ve	11	11	-ve
8	CoPCplx	14	10	7	12	9	7	10	9	-ve	11	9	-ve
9	DMSO	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
<u> </u>	(Control)												

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S/No	Compound	Samonella entrica			Esherichia coli			Staphylococcus aureus			Bacillus subtilis		
		Concen	tration (n	ng/ml)	Concer	ntration (n	ng/ml)	Concentration (mg/ml)			Concentration (mg/ml)		
		50	25	12.5	50	25	12.5	50	25	12.5	50	25	12.5
1	½LAN	10	7	-ve	7	-ve	-ve	-ve	-ve	-ve	8	-ve	-ve
2	½LOA	8	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
3	LAN	8	7	7	-ve	-ve	-ve	-ve	-ve	-ve	11	-ve	-ve
4	LOA	8	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	10	-ve	-ve
5	FeOCplx	10	9	9	11	9	-ve	12	10	8	12	11	7
9	FeNCplx	16	14	10	14	12	8	13	11	8	14	12	9
10	CoOCplx	14	10	8	15	13	9	14	10	9	14	11	9
11	CoNCplx	14	11	10	15	12	9	14	12	9	15	13	9
12	DMSO	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
	(Control)												

Table 6.0: Result of the Antibacterial Activities of Ligands and their Metal Complexes

Diameter of Inbition Zone (mm)

## 3. Results and Discussion

The ligands and complexes are stable at room temperature; maintain their characteristic coloration, non hygroscopic and soluble in common organic solvents like ethanol, acetone, methanol and Dimethyl Sulphoxide (DMSO) but insoluble in water. The physical properties, molar conductivity and elemental composition are shown in table 1.0.

The progress of the reactions was monitored with infra red (IR) spectroscopy (table 2.0). The reaction of 2hydroxynaphthalene-1-carbaldehyde and the two substituted aniline show that, stretch due to primary amine v (N-H) was observed only on half unit reactions of 1/2 LNA (3414.764) cm<sup>-1</sup> and <sup>1</sup>/<sub>2</sub> LOA (3495.558) cm<sup>-1</sup> and disappeared in Schiff base ligands and complexes due to utilization of the amino protons in bond formation. Likewise, the v (OH) absorptions were recorded on the ligands within the region of (3262.959-333.483) cm<sup>-1</sup> and was not observed on complexation because of the involvement of oxygen in formation of metallic bonding [6, 13, 14] and non participating of OH group in bonding

[15]. The IR absorption of v (C=N) of the ligands show increasing values when compared to their corresponding complexes, the highest value v (C=N) recorded <sup>1</sup>/<sub>2</sub>LOA (1627.608) cm<sup>-1</sup> followed by that of FeNcplx (1621.397) cm<sup>-1</sup>. This value shifted as low as 1572.93 cm<sup>-1</sup> being that of CoOcplx. The phenomenon is attributed to the coordination of azomethine nitrogen to the metal ion [14, 16]. The absorption of phenolic oxygen v (C-O) were recorded in all the metallic complexes indicating coordination of the phenolic oxygen with metal ions through deprotonation [10] with the lowest value recorded CoNcplx (1259.849) cm<sup>-1</sup>. There are evidence of the formation of M-N and M-O bonds in all the complexes with their values lower than their corresponding ligands, ranging from (790.196-887.106) cm<sup>-1</sup> for v (M-N) and (738.013 - 790.196) cm<sup>-1</sup> for v (M-O) respectively. These high value opposed to results recorded [15, 14] but agreed with [7, 22]. This irregularity in the assignment of bands in the far-IR region to metal bonds has been described as controversial [15] and purely tentative [10] because of various skeletal vibrations associated with metal ligand bonding.

Compounds / ligonds	Must	Colour	Mpt	Yield	$\Omega^{-1}$ cm <sup>2</sup> mol <sup>-1</sup> Molar Cond.		Elemental Composition %)			
Compounds / ligands	<b>W1.W</b> t	Colour	<sup>0</sup> C	%	In acetone	In ethanol	С	Н	Ν	0
$C_{17}H_{14}N_2O~(\frac{1}{2}~LNA)$	262	Light Brown	148-150 <sup>0</sup> C	80	3.4	1.5	67.60	6.70	25.40	-
C <sub>17</sub> H <sub>13</sub> NO <sub>2</sub> (½LOA)	259	Orange	215-217 <sup>0</sup> C	85	4.1	1.2	67.97	5.99	12.44	13.60
$C_{28}H_{20}N_2O_2$ (LNA)	416	Black	180-182°C	60	4.5	3.3	50.48	2.91	6.01	6.79
$C_{28}H_{21}NO_3$ (LOA)	419	Dark Brown	250-352 <sup>0</sup> C	85	3.1	0.9	64.20	4.15	10.20	6.88
FeC <sub>28</sub> H <sub>18</sub> N <sub>2</sub> O <sub>3</sub> FeOCplx	443	Light Brown	300≤0 <sup>0</sup> C	60	2.5	2.3	52.90	3.96	8.08	12.26
FeC <sub>28</sub> H <sub>18</sub> N <sub>2</sub> O <sub>2</sub> FeNCplx	440	Grayish Brown	280-282°C	65	5.2	1.8	39.80	3.40	4.62	15.91
CoC <sub>28</sub> H <sub>20</sub> NO <sub>3</sub> CoOCplx	444	Blackish Brown	280≤0 <sup>0</sup> C	69	10.7	7.5	55.80	4.24	8.41	12.90
CoC <sub>28</sub> H <sub>18</sub> N <sub>2</sub> O <sub>2</sub> CoNCplx	441	Brown	250-252 <sup>0</sup> C	80	9.1	8.4	39.40	2.39	4.28	18.20

**Table 1.0:** Physical and analytical data of half ligands, Schiff base, metal complexes and their elemental composition

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## KEY:

 $\frac{1}{2}$  LNA = Product of 2-hydroxynophthalene-1carbaldehyde and 2-aminoaniline =  $\frac{1}{2}$  unit ligands of 2aminoaniline

 $\frac{1}{2}$  LOA = product of 2-hydroxynaphthalene-1carbaldehyde and 2-hydroxyaniline =  $\frac{1}{2}$  unit ligands of 2hydroxyaniline

LNA = Schiff base of 2-aminoaniline

LOA = Schiff base of 2-hydroxyaniline

Fe OCPLX = Iron (II) complex of Schiff base of 2hydroxyaniline

Fe NCPLX = Iron (II) complex of Schiff base of 2aminoaniline Co OCPLX = Cobalt (II) complex of Schiff base of 2-

hydroxyaniline Co NCPLX = Cobalt (II) complex of Schiff base of 2aminoaniline

Table 2.0: IR results of half ligands, Schiff bases and their metallic complexe	s
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Compounds: ( <sup>1</sup> /2ligands, Schiff and Complexes)	V (N-H) cm-1	V (OH) cm-1	V (C =N) cm-1	V (C-O) cm-1	V (M-N) cm-1	V (M-O) cm-1
<sup>1</sup> / <sub>2</sub> LNA (C <sub>17</sub> H <sub>14</sub> N <sub>2</sub> O)	3414.764	3321.965	1613.332	-	-	-
<sup>1</sup> / <sub>2</sub> LOA (C <sub>17</sub> H <sub>13</sub> NO <sub>2</sub> )	3495.558	3330.483	1627.608	-	-	-
LNA (C <sub>28</sub> H <sub>20</sub> N <sub>2</sub> O <sub>2</sub> )	-	3262.959	1621.198	-	-	-
LOA (C <sub>28</sub> H <sub>21</sub> N O <sub>3</sub> )	-	3321.222	1618.179	-	-	-
FeOCplx FeC <sub>28</sub> H <sub>20</sub> NO <sub>3</sub>	-	-	1617.665	1267.295	790.196	738.013
F e NCplx (Fe C <sub>28</sub> H <sub>18</sub> N <sub>2</sub> O <sub>2</sub> )	-	-	1621.392	1274.750	887.106	790.196
CoNCplx CoC <sub>28</sub> H <sub>18</sub> N <sub>2</sub> O <sub>2</sub>	-	-	1572.930	1263.568	820.014	738.013
CoOCplx CoC <sub>28</sub> H <sub>20</sub> NO <sub>3</sub>	-	-	1599.028	1259.840	812.560	741.740

 Table 3.0: UV-VIS absorption bands of half ligands, Schiff bases and metal complexes

c	Samples	UV-VIS Absorbtion					
S∕NO	Samples	(200-880 nm)					
1	Half unit ligands of 2-aminoaniline (1/2 LNA)	575 nm					
2	Half unit ligands of 2-hydroxyaniline (1/2 LOA)	580 nm					
3	Schiff base of 2-aminoaniline (LNA)	575, 590, 760 nm					
4	Schiff base of 2-hydroxyaniline (LOA)	478 nm					
5	Fe C <sub>28</sub> H <sub>20</sub> N O <sub>3</sub> (Fe OCPLX)	584 nm					
6	Fe $C_{28} H_{28} H_{18} N_2 O_2$ (Fe NCPLX)	578, 592 nm					
7	Co C28 H20 N O3 (Co OCPLX)	600, 605 nm					
8	Co C28 H18 N2 O2 (Co NCPLX)	560, 590 nm					

Table 4.19: Summary of Mass spectrometric Results of Half Ligands and their fragments

m/z	Assignment/Formula of compound	Structure fragments
262.33	<sup>1</sup> ⁄ <sub>2</sub> LNA-Half ligand of 2-aminoaniline	C <sub>17</sub> H <sub>14</sub> N <sub>2</sub> O (ion molecule)
170	C11H8NO	N OH

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473.48	FeOCplx- Iron(II) Complex of Schiff	FeC <sub>28</sub> H <sub>19</sub> NO <sub>3</sub>
	base of 2-hydroxyaniline	(Ion Molecule)
119	C7H5NO	$H_{HC=N}$ O
		-0-
172	C11H8O2	·O· C=N
168	C11H6NO	



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Key: FeNACplx-Iron (II) Complex of Schiff base of 2-aminoaniline

FeOACplx-Iron (II) Complex of Schiff base of 2-hydroxyaniline

CoNACplx – Cobalt (II) Complex of Schiff base of 2-aminoaniline

CoOACplx-Cobalt (II) Complex of Schiff base of 2-hydroxyaniline

The UV-VIS absorption of ligands and complexes are greatly affected if such substances contain chromophoric functional groups like the double bonds or tripple bonds, benzene rings, carbonyl and thiocarbonyls groups. This absorption can be enhanced further if there is the presence of auxochrome like OH, NH2, CH3, NO2, etc which do not absorb significantly in the UV region but have effects on the molecules to which they attached. These causes absorption to longer wavelength (low energy) or red shift related to electron-donating effect [19]. The UV-VIS absorption of the ligands and complexes (table 3.0) show characteristics behavior in their peaks with LNA having three peaks (575, 590 and 760) nm. The highest absorption of the complexes is recorded on CoOCPlx with two peaks at (600.605) nm. This splitting of peaks is due to excitation of different electronic, vibrational and rotational transitions [20] and are attributed to  $n-\pi^*$  and  $\pi$ **π**\* [21].

The results of mass spectrometry of the compound are shown in (table 4.0), depicting the m/z results of the ligands and complexes as well as that of the fragments. These m/z results confirmed the structure of the half ligands, Schiff base and complexes. For instance the m/z of  $\frac{1}{2}$  LNA (262.33,  $\frac{1}{2}$  LOA (236.31), LNA (416.5) and LOA (419.5). while that of metal complexes FeNCplx (470.48), FeOCPlx (473.48), CoNCPlx (473.49) and CoOCPlx (476.41). The mass/charge of the fragments are illustrated in table 4.0.

The microbial activity of ligands and complexes were tested against selected bacteria: *Salmonella enterica* (G -), *Eschericha coli* (G -), *Staphylococcus aureus* (G+) and *Bacillus subtilis* (G+), and selected fungi: *Aspergillus flavus, Fusarium moniliforme, Rhizopus stolonifer* and *Fusarium solani*. The results (Tables 5.0 and 6.0) show that, the metal complexes exhibit more activity on test

organism than the ligands [18, 14] and the effects are more pronounced in antibacterial than in antifungal activities as shown on the area of diameter of inhibition zone. The effectiveness of complexes is attributed to chelation, delocation of pii-electrons of chelate ring and lipophilicity of the complexes leading to the breakdown of permeability barrier of cell organism thereby retarding growth process [14, 23].

## 4. Conclusion

Iron (II) and Cobalt (II) complexes of Schiff base derived from 2-hydroxynaphthalene-1-carbaldehyde using 2aminoaniline and 2-hydroxyaniline were prepared, synthesized and characterized. The ligands and complexes are stable, maintain their characteristics coloration and soluble in organic solvents. The IR results depicts the progress of the reaction and chelation as evident in the disappearances of v (N-H) of the primary amine and v (OH) of phenolic hydroxide because of the involvement of oxygen in complexation. The UV-VIS results of the test compound show characteristic behaviors in their peaks (table 3.0) while the mass spectroscopy illustrated the m/z of the compounds authenticating their structures and molecular weights. The metallic complexes show antimicrobial effectiveness when compare to the ligands.

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## **Data Availability**

The data supplied in this manuscript are available and will be made available on request.

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