

Synthesis, Characterization and Antimicrobiological Activity Studies of Cobalt (II) and Nickel (II) Complexes Derived From 2-Hydroxynaphthalene-1-Carbaldehyde Using 2-Thiosylaniline and 2-Aminoaniline As Co-Schiff Bases.

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Abstract

Four complexes CoScplx, CoNcplx, NiScplx and NiNcplx were synthesized from 2-hydroxynaphthalene -1-carbaldehyde using 2-thiosylaniline and 2-aminoaniline as Co-Schiff bases. The physical and analytical data of the ligands and complexes such as Molecular weight, colour, melting point and percentage yield as well as molar conductivity and elemental composition were determined. The compounds were characterized using FT-IR, UV-VIS and MS Analyzers. FT-IR results revealed loss of -OH bond noticed in the ligands at complexation due to deprotonation and utilization of phenolic OH in bond formation. The UV-VIS spectral data recorded more peaks with Schiff bases and complexes with 2-aminoaniline as a result of more chromophoric groups. The MS result determined m/z of the molecular ions and their fragments confirming the structure of synthesized compounds. The complexes demonstrated more antifungal and antibacterial activities more than the ligands hence can be incorporated into antibiotics drugs in future.

Key Words: Synthesis, Characterization, Antimicrobial, 2-thiosylaniline, 2-aminoaniline.

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I. Introduction

Spectrophotometry has been a fundamental tool for determination of concentration of elements based on laws of photometry [1]. It helps in the determination of configuration and geometry of compound by ultraviolet (UV) [2], structural analysis by infrared [3], identify, quantify and elucidate structure of known and unknown compounds by mass spectrometry[4,5]. Schiff bases have gained a lot of interest in both the pharmaceutical and medical field [6] and are very popular ligands because of their easy formation and valuable coordination characteristic[7,8]. Metal complexes formed from 2-hydroxynaphthalene-1-carbaldehyde have biological effectiveness [9,11] and the ligands coordinate to metal ions through azomethine nitrogen (C=N) [10]. Aniline and its substituents: 2-aminoaniline and 2-thiosylaniline are very effective in synthesis and can coordinate at N,S during complexation [9,12,14] and show greater activity against micro organism[13]. Cobalt and Nickel complexes play important role in the field of medicine, bioinorganic chemistry, functional materials as well as antimicrobial activities [15] and regulation of DNA synthesis [16,17]. This study: synthesis, characterization and antimicrobial activity of cobalt(II) and Nickel(II) complexes derived from 2-hydroxynaphthalene-1-carbaldehyde using 2-thiosylaniline and 2-aminoaniline tried to evaluate these compounds using spectral (IR, UV-VIS and MS) means with the view to determining its anti microbial efficacy.

II. Experimental

2.1 Materials and electronic equipments

The reagents used included β -hydroxynaphthalene-1-carbaldehyde (Sigma Aldrich (SA)), 2-aminoaniline (J-T. Baker (JTB)), 2-thiosylaniline(SA), and Salicylaldehyde (SA). Others were Cobalt(II)acetate tetrahydrate (JTB), Nickel(II)acetate tetrahydrate (British Drug House(BDH), Piperidine(JTB) and

Dimethylsulphoxide(DMSO)(SA). The solvents included Ethanol (GH Tech), Methanol (JTB), Acetone (JTB) and Ether (SA).

The electronic equipments: Golden mettle USA Electronic balance model 3002, Magnetic Stirrer Hot plate 7a-1 Techmel & Techmel USA, J.J.I Precision Force Electronic mixer (China), melting point apparatus, HACH HC40d conductivity meter, IR-Fourier Transformed infrared spectrophotometer, UV-VIS-Spectrum lab 725s UV-VIS spectrophotometer, CE-440 Elemental Analyzer(Exeter Analytical Inc, UK) Agilent 6890A gas chromatograph, 5973C Inert Mass Spectrometer (Agilent Technologies).

For antimicrobial analysis: Incubator Desktop constant temperature –E- track scientific instruments, England, Autoclave –E- track scientific instrument, England, Microscope- OPTIKA Italy, Electronic weighing balance- Ohaius cooperation, pine Brook, N.J. USA; SDA- live saver (Lis) Biotech San Diego CA 92121, USA; Muller Hinton agar-Titan Biotech Ltd, Rajasthem, India and Nutrient Agar TM media, Titan Biotech Ltd, Rajasthem, India.

Micro organisms: Fungi: - *Aspergillus flavus*, *Fusarium moniliforme*, *Rhizopus stolonifer* and *Fusarium solani*. Bacteria: *Salmonella enterica*, *Esherichia coli*, *staphylococcus aureus* and *Bacillus subtilis*.

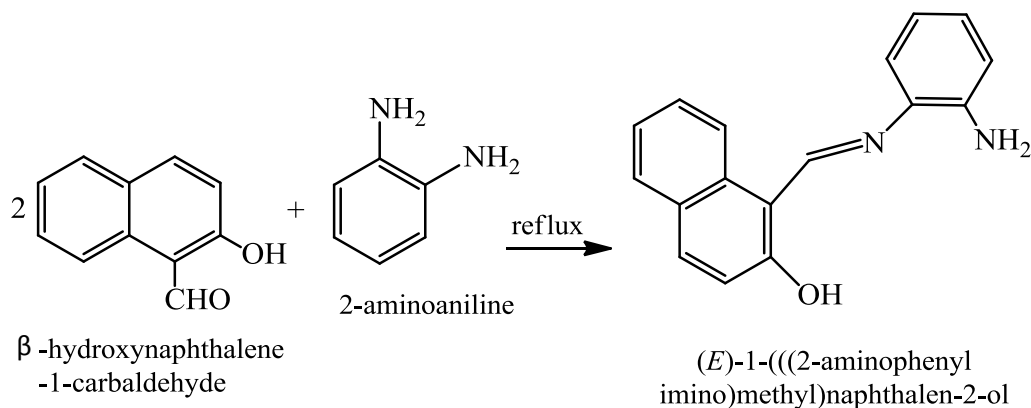
2.2 Synthesis

The synthetic methods adopted were reported by the following literature [18, 19]

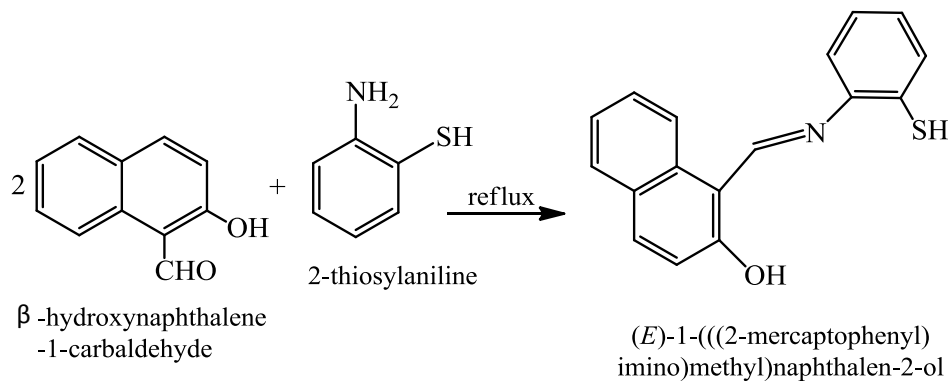
EQUATION OF REACTIONS:

2.2.1 Preparation of Half Unit ligands (½LNA, ½ LSA)

A solution of each of the two anilines (2.5g of 2-thiosylaniline or 2.16g of 2-aminoaniline, that is, 20 mmol each) was dissolved with 150ml of ethanol in a 500 ml round bottomed flask. To this was added drop wise with vigorous stirring 6.89g (20 mmol) of 2-hydroxynaphthalene-1-carbaldehyde dissolved in 100ml anhydrous ethanol. The two solutions were stirred for 30 minutes and then reflux for 30minutes. The resulting solution was evaporated in vacuum to remove the solvent and the solid residue washed with cold ethanol and then recrystallized with a mixed solvent (1:1:1) ratio of methanol, ethanol and Acetone [19]. After drying, the melting point and percentage yield were determined.



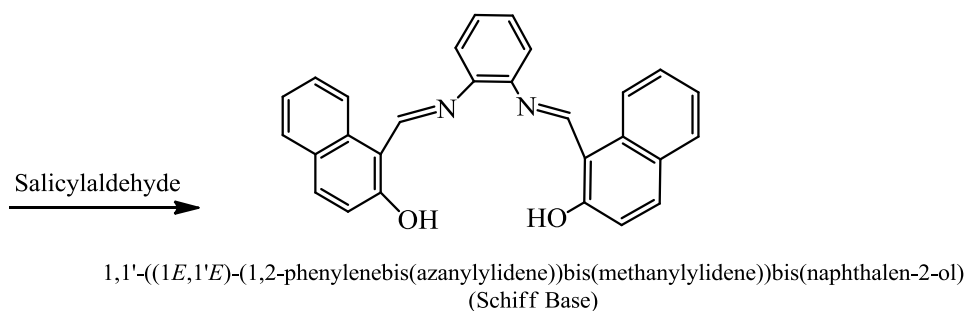
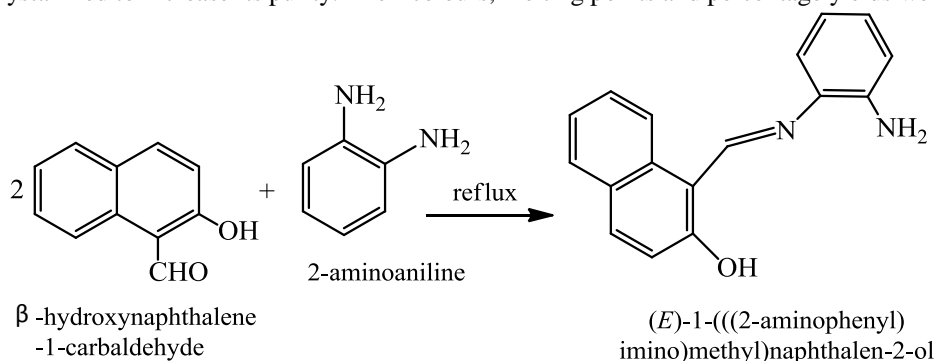
Scheme 1: Preparation of ½LNA Half Unit ligand



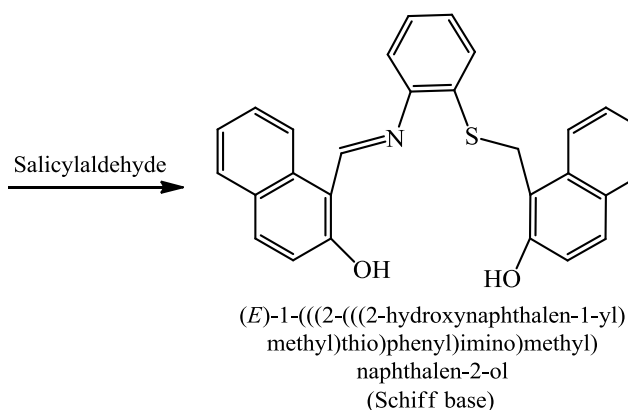
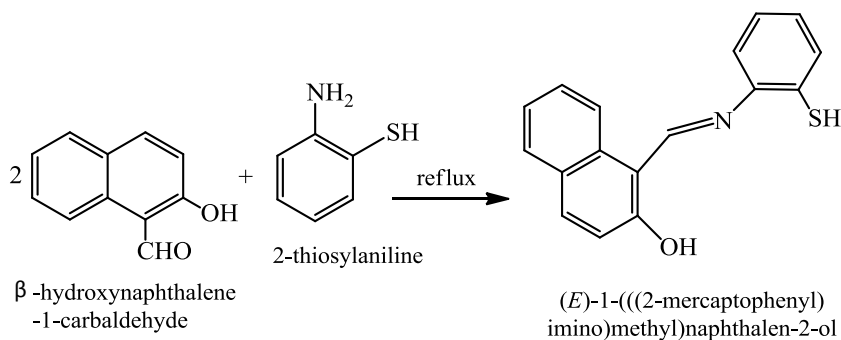
Scheme 2: Preparation of ½LSA Half Unit ligand

2.2.2 Preparation of Schiff base ligands (LNA, LSA)

A solution of each of the half unit Ligands ($\frac{1}{2}$ LNA, 2.62g or $\frac{1}{2}$ LSA, 2.79g) was prepared in anhydrous ethanol and a solution of 10 mmol (1.22g) of salicylaldehyde. The two solutions were mixed and refluxed for 1 hour. The resultant product was concentrated in vacuum, in it precipitated coloured crystals. It was further recrystallized to increase its purity. Their colours, melting points and percentage yields were determined.



Scheme 3: Preparation of LNA Schiff Base Ligand

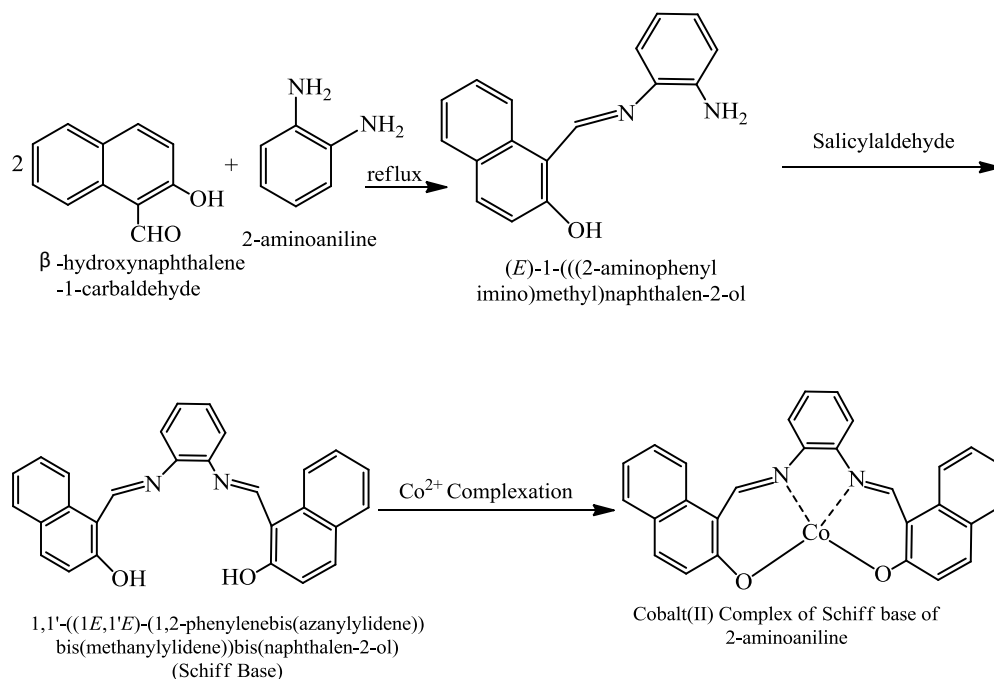


Scheme 4: Preparation of LSA Schiff Base Ligand

2.2.3 Preparation of Co(II) and Ni(II) complexes

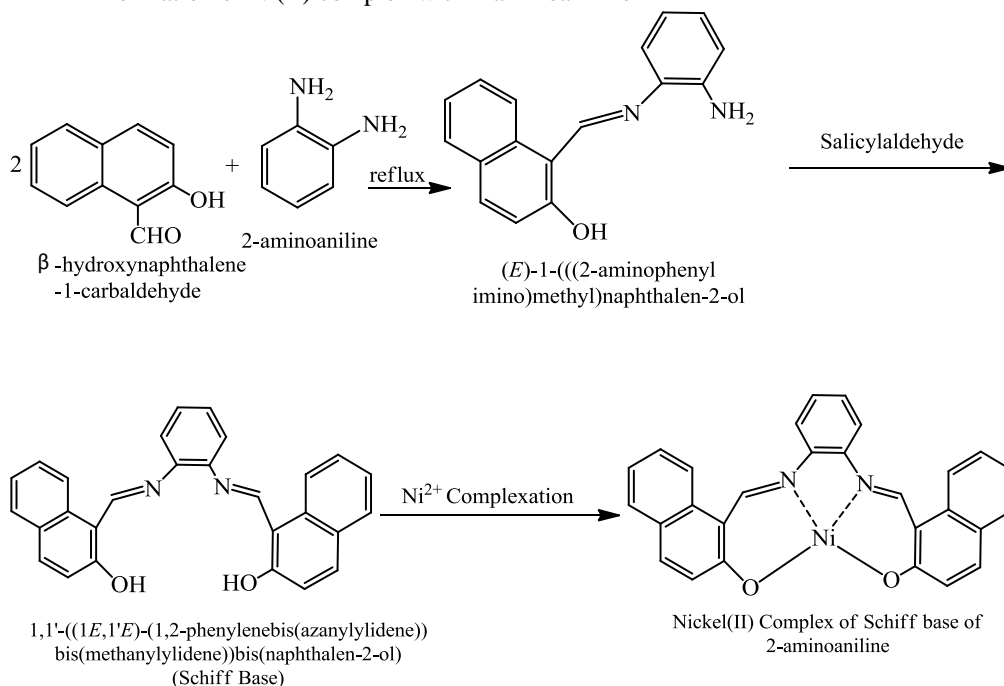
A 1 mmole each of the metal acetate (Co(II), 0.217g; Ni(II), 0.218g) was placed in boiling methanol solvent. 1 mmole solution of the Schiff base ligands (LNA 0.416g, LSA 0.435g) was added, then few drops of piperidine were also added as a precursor. The whole mixture was refluxed for 1 hour. After refluxing, it was then allowed to stand to deposit coloured crystals. The precipitate was washed with the cold methanol and cold ether and allowed to dry in an oven at a temperature of 50°C

(a) Formation of Co(II) complex with 2-aminoaniline



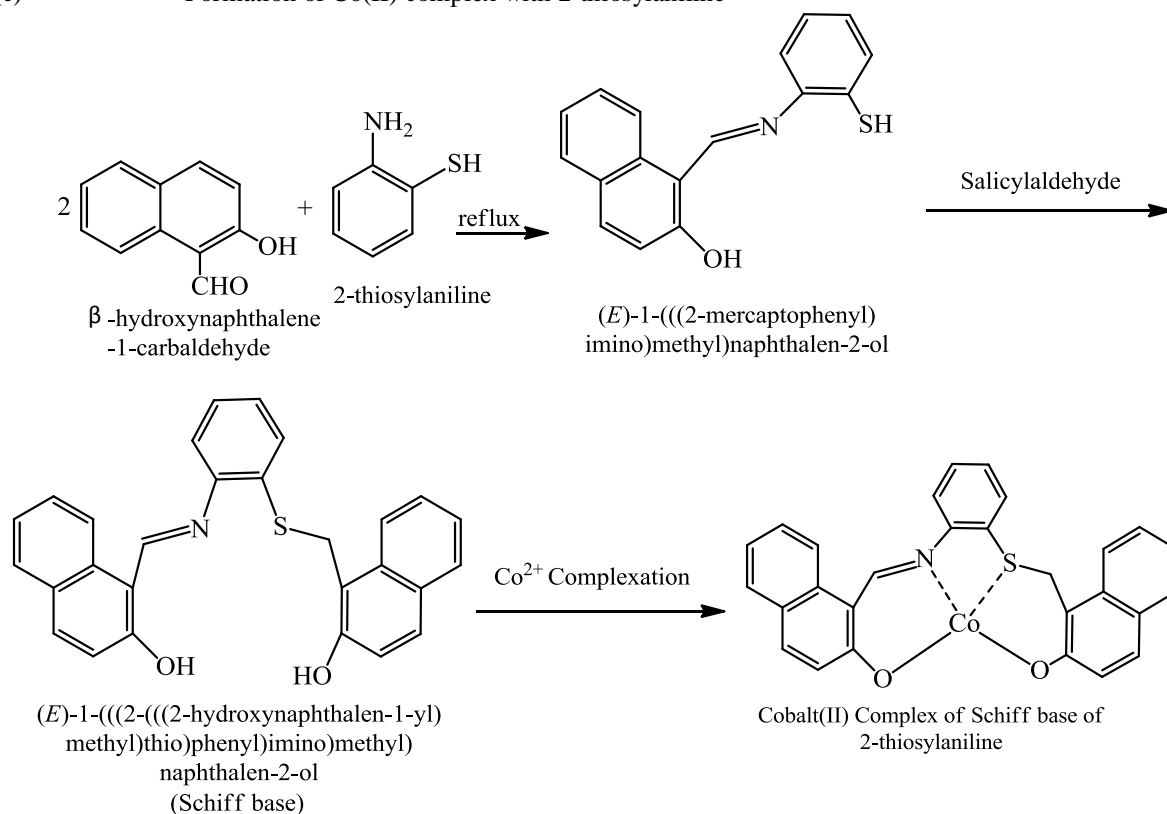
Scheme 5: Preparation of Co(II) Complex with 2-aminoaniline

(b) Formation of Ni(II) complex with 2-aminoaniline



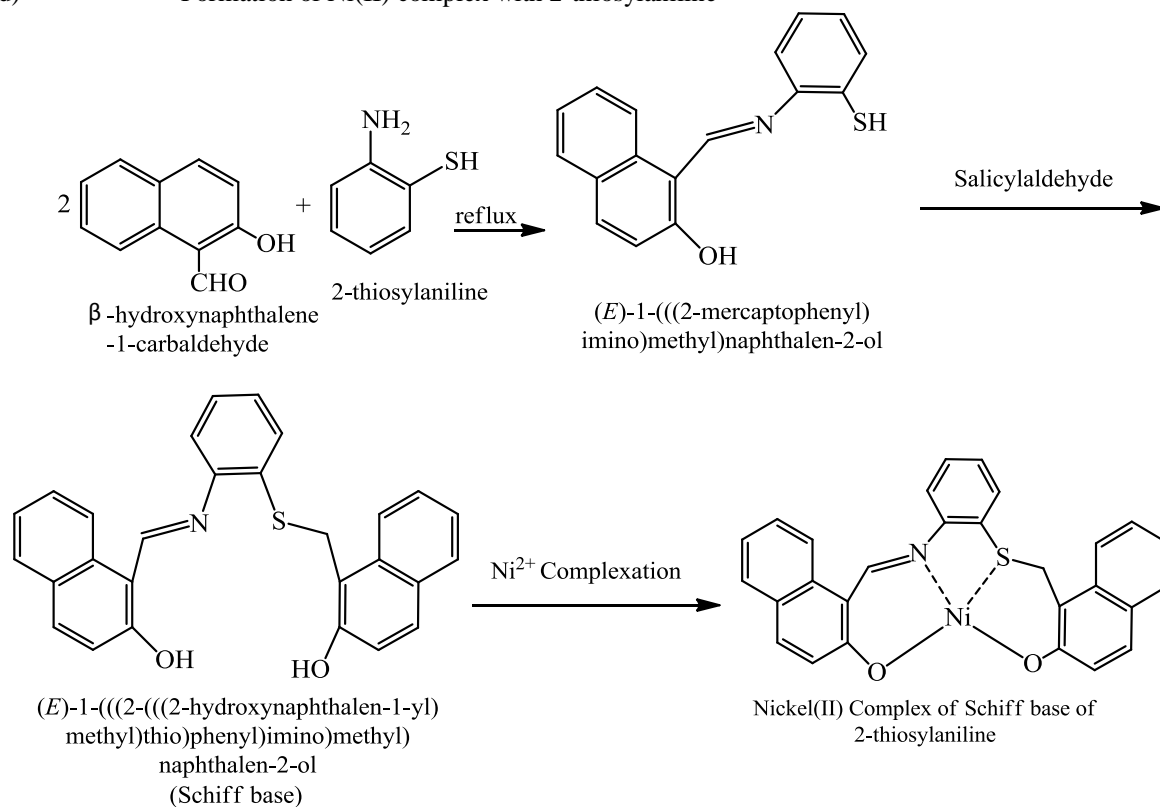
Scheme 6: Preparation of Ni(II) Complex with 2-aminoaniline

(c) Formation of Co(II) complex with 2-thiosylaniline



Scheme 7: Preparation of Co(II) Complex with 2-thiosylaniline

(d) Formation of Ni(II) complex with 2-thiosylaniline



Scheme 8: Preparation of Ni(II) Complex with 2-thiosylaniline

2.2.4 Elemental And Spectral Determination

These were determined using the electronic equipments mentioned above (section 2.1) carried out in ISI analytical laboratory, Surulere Lagos, Nigeria [20]

2.3 Determination Of Antimicrobial Activities

The antimicrobial activities were determined using the following standard methods [10, 21, 22]

2.3.1 Procedure For Antibacterial Test

The antibacterial activity of the compounds were evaluated using the paper disc diffusion method against different species of bacteria: *Salmonella enterica* (G⁻), *Escherichia coli* (G⁻), *Staphylococcus aureus* (G⁺) and *Bacillus subtilis* (G⁺)

The test compounds were dissolved in DMSO solution from where different two fold serial diluted concentrations were obtained (50 mg/ml, 25 mg/ml and 12.5 mg/ml) from the stock solution. A muller Hinton agar plate was prepared using sterile cotton swabs. The plates were seeded with the test micro-organism diluted to 0.5 McFarland Standards (in 10⁸ CFU/ml). Therefore, paper disc previously impregnated with different concentrations of the test compound was aseptically placed on the plates. DMSO was also placed in the plates as negative control. The plates were incubated at 35⁰C for 16-18 hours. The antibacterial activity was evaluated by measuring the diameter of inhibition zone around the disc to the nearest millimeter (mm).

2.3.2 Procedure For Antifungal Activity Test

Paper disc diffusion method was also used against different fungal species: *Aspergillus flavus*, *Fusarium moniliforme*, *Rhizopus stolonifer* and *fusarium solani*. Sterile savorands dextrose agar (SDA) plates were inoculated with fungal suspension prepared from a 5-day evenly spread over the medium using cotton swabs.

Therefore, paper disc impregnated with different concentrations (50 mg/ml, 25 mg/ml and 12 mg/ml) of the test compounds were aseptically placed using forcep. The plates were observed for formation of clear inhibition zone around the disc which indicated antifungal activity. The diameter of inhibition zones were measured to the nearest millimeter (mm).

3.0 Results And Discussion

The physical and analytical data of half unit and the Schiff base ligands as well as their metal complexes (scheme1.0) are shown on table 1.0

Table 1.0 Physical and Analytical data of ligands and complexes

Ligands/Compound	M.wt	Colour	Mpt	Yield %	$\Omega^{-1}\text{cm}^2 \text{mol}^{-1}$		Elemental Composition (%)				
					Molar Cond.		C	H	N	S	O
					In acetone	In ethanol					
C ₁₇ H ₁₄ N ₂ O (½ LNA)	262	Light Brown	148-150°C	80	3.4	1.5	67.60	6.70	25.40	-	-
C ₁₇ H ₁₃ N ₂ O (½ LSA)	279	Yellow	78-80°C	79	2.7	1.4	57.72	5.99	11.05	25.57	-
C ₂₈ H ₂₁ NSO ₂ (LSA)	435	Orange Yellow	102-104°C	80	2.5	1.0	50.91	4.60	18.34	20.80	5.18
C ₂₈ H ₂₀ N ₂ O ₂ (LNA)	416	Black	180-182°C	60	4.5	3.3	65.48	7.91	19.01	-	6.79
CoC ₂₈ H ₁₈ N ₂ O ₂ (CoNcplx)	441	Brown	250-252°C	80	9.1	8.4	39.40	2.39	4.28	-	18.20
CoC ₂₈ H ₁₈ NSO ₂ (CoScplx)	460	Black	300≤0°C	70	3.6	1.7	36.82	3.30	13.20	15.11	3.79
NiC ₂₈ H ₁₈ N ₂ O ₂ (NiNcplx)	442	Light Brown	280≤0°C	85	4.1	5.1	45.42	2.24	5.30	-	6.10
NiC ₂₈ H ₁₈ NSO ₂ (NiScplx)	461	Orange	285-287°C	75	4.0	8.5	40.95	3.69	14.72	16.82	8.41

Key: ½ LNA = Half unit ligand of 2-aminoaniline
 ½ LSA = Half unit ligand of 2-thiosylaniline
 LNA = Schiff base ligands of 2-aminoaniline
 LSA = Schiff base ligands of 2- thiosylaniline
 CoNcplx = Cobalt(II) complex of 2-aminoaniline
 CoScplx = Cobalt(II) complex of 2- thiosylaniline
 NiNcplx = Nickel(II) complex of 2-aminoaniline
 NiScplx = Nickel(II) complex of 2-thiosyaniline

3.1 Physical Properties

The synthesized complexes show typical colorations and are stable at room temperature appears as powder at high temperature[23] and non hygroscopic. Their melting points were determined as well as their percentage yield (Table1.0). It was observed that, the melting points of ligands were lower than their corresponding metal complexes indicating chelation. Both ligands and complexes are soluble in organic solvents: Ethanol, acetone, methanol and DMSO and the observed molar conductance values both in acetone and ethanol are too low(1.0-9.1 $\Omega^{-1}\text{cm}^2\text{mol}^{-1}$) indicating non-electrolytic nature[12,24,25]

Table 2.0 FT-IR spectral results of ligands and their metal complexes

Ligands And complexes	$\nu(\text{N-H})$ cm^{-1}	$\nu(\text{C=N})$ cm^{-1}	$\nu(\text{OH})$ cm^{-1}	$\nu(\text{SH})$ cm^{-1}	$\nu(\text{C-O})$ cm^{-1}	$\nu(\text{C-S})$ cm^{-1}	$\nu(\text{M-N})$ cm^{-1}	$\nu(\text{M-S})$ cm^{-1}	$\nu(\text{M-O})$ cm^{-1}
½ LNA	3414.764	1613.332	3321.965	-					
½ LSA	3498.395	1618.179	3321.222	2626.556					
LNA	-	1621.198	3262.959	-					
LSA	-	1610.888	3167.589	2540.346					
CoNcplx	-	1599.028	-	-	1259.840	-	812.560	-	741.740
CoScplx	-	1595.301	-	-	1263.568	1457.389	820.014	879.652	741.740
NiNcplx	-	1602.755	-	-	1282.204	-	816.287	-	741.740
NiScplx	-	1610.210	-	-	1308.296	1431.298	820.014	887.106	738.013

3.2 FT-IR Spectra Analysis

The summary of the spectral results of FT-IR data of the ligands and complexes are shown on (Table 2.0). The observed $\nu(\text{N-H})$ of primary amines were recorded on half unit ligands (½LNA 3414.764 cm^{-1} and ½ LSA 3498.395 cm^{-1} and absent in Schiff base ligands and complexes due to loss of the proton during complexation[19]. The absorption band $\nu(\text{OH})$ were noticed in the ligands but were absent in complexes because its involvement in complexation and reaction with metal(II) through deprotonation [12,19,26] and non participation of OH group in bonding [24]. The thionyl proton $\nu(\text{SH})$ was recorded only on ½ LSA (2622.550 cm^{-1}) and LSA (2540.346 cm^{-1}) and this manifested at a lower absorption ranges in $\nu(\text{C-S})$ and $\nu(\text{M-S})$ showing participation of $\nu(\text{C-S-C})$ and $\nu(\text{C-S-M})$ bonding in the complexation processes[27]. The absorption band due to phenolic oxygen $\nu(\text{C-O})$ were recorded in all the M(II) complexes indicating coordination of phenolic oxygen with metal ions through deprotonation[28]. The $\nu(\text{M-S})$ and $\nu(\text{M-O})$ bands ranges from (812.560-820.014) cm^{-1} and (738.01-740) cm^{-1} respectively. These high values opposed to low ranges (445-581 cm^{-1}) recorded by these authors [24,26] but agrees with high values (695-770 cm^{-1}) of [13,29,30] and 1318 cm^{-1} [31]. This irregularity in the assignment of bands in the far-infrared region to metal bonds has been described as controversial [24] and tentative [28] because of various skeletal vibrations associated with metal-ligands complexes.

3.3 UV-VIS Analysis

Table 3.0 UV-VIS and Conductivity results of the samples

S/No	Ligands and M(II) complexes	Conductivity $\Omega^{-1}\text{cm}^2\text{mol}^{-1}$		UV-VIS (200-800nm)
		In Acetone	In Ethanol	
1	½ LNA	3.4	1.5	575
2	½ LSA	2.7	1.45	584
3	LNA	4.5	3.3	575,590,760
4	LSA	2.5	1.0	584,598
5	CoNcplx	9.1	8.4	560,590
6	CoScplx	3.6	1.7	680
7	NiNcplx	4.1	5.1	573,586
8	NiScplx	4.0	8.5	586

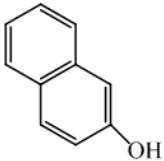
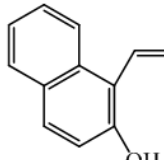
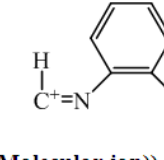
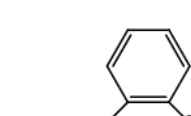
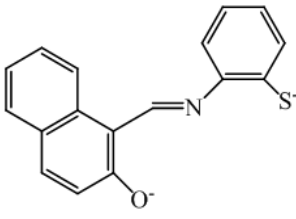
The absorption of ligands and their complexes are greatly affected if such substances contain chromophoric functional groups like double or triple bonds, benzene rings, carbonyl and thiocarbonyl groups. This absorption can be enhanced further if there is the presence of auxochromes like OH, NH₂, CH₃, NO₃ etc which do not absorb significantly in UV regions but have effect on the molecules to which they are attached. This causes absorption to longer wavelength (low energy) or red shift related to electron donating effect [32]. The results of absorption (Table 3.0) show characteristic behavior in the number of peaks and falls within visible region (380-800 nm) of the UV-VIS spectrometer. The Schiff base ligands of 2-aminoaniline show three peaks at 570, 590 and 760 nm and reduced to two peaks or one at their respective metal complexes due to utilization of the OH in bond formation. Presence of functional group causes splitting of peaks due to excitation of different electronic, vibrational and rotational transitions[33] hence the peaks are attributed to n→π* and π→π* transitions

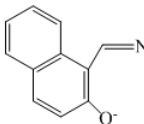
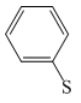
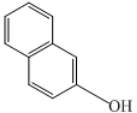
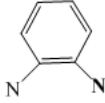
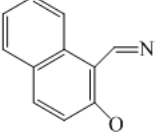
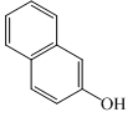

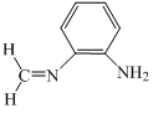
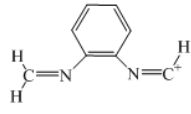

3.4 Mass Spectrometry Results

The Mass Spectrometry (MS) of the results show mass to charge (m/z) values of the samples. It depicts the molecular weight of the predicted structures (schemes 1-8) of the ligands and complexes as well as that of fragments (Table 4.0) thereby authenticating the reaction processes of the synthesis. The MS of the results are summarized in the table 4.0

Table 4.0 summary of Mass spectrometry of ligands, complexes and their fragments

Table 4.0 Summary of Mass spectrometry of ligands, complexes and their fragments

m/z	Assignment/Formula of compound	Structure fragments
262.33	½ LNA- C ₁₇ H ₁₄ N ₂ O	(Molecular ion) 
144	C ₁₀ H ₈ O	
170	C ₁₁ H ₉ NO	
119	C ₇ H ₇ N ₂	
279.37	½ LSA – C ₁₇ H ₁₂ NSO	(Molecular ion))
277	C ₁₇ H ₁₇ NOS	

169	$C_{11}H_7NO$	
109	C_6H_5S	
416.5	LNA- $C_{23}H_{20}N_2O_2$	(Molecular ion)
144	$C_{10}H_8O$	
104	$C_6H_4N_2$	
435.56	LSA- $C_{23}H_{21}NSO_2$	(Molecular ion)
168	$C_{11}H_6NO$	
144	$C_{10}H_8O$	
122	C_6H_4NS	
473.41	CoNcplx - $CoC_{28}H_{18}N_2O_2$	(Molecular ion)
119	$C_7H_7N_2$	
131	$C_8H_7N_2$	
104	$C_6H_4N_2$	
476.41	CoSpplx - $CoC_{28}H_{19}NSO_2$	(Molecular ion)

134	C ₇ H ₄ NS	
105	C ₇ H ₆ N	
104	C ₇ H ₆ N	
473.19	NiNcplx – NiC ₂₈ H ₁₈ N ₂ O ₂	(Molecular ion)
169	C ₁₁ H ₇ NO	
104	C ₇ H ₆ N	
492.92	NiScplx – NiC ₂₈ H ₁₉ NSO ₂	(Molecular ion)
143	C ₁₀ H ₇ O	
122	C ₈ H ₄ NS	
135	C ₇ H ₅ NS	

Table 5.0 Results of the Antibacterial Activities of Ligands and their Metal (II) Complexes

Diameter of Inhibition Zone (mm)

S/No	Ligands and complexes	<i>Salmonella enterica</i> Concentration (mg/ml)			<i>Esherichia coli</i> Concentration (mg/ml)			<i>Staphylococcus aureus</i> Concentration (mg/ml)			<i>Bacillus subtilis</i> Concentration (mg/ml)		
		50	25	12.5	50	25	12.5	50	25	12.5	50	25	12.5
1	½LNA	10	7	-ve	7	-ve	-ve	-ve	-ve	-ve	8	-ve	-ve
2	½LSA	8	7	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
3	LNA	8	7	7	-ve	-ve	-ve	-ve	-ve	-ve	11	-ve	-ve
4	LSA	7	7	-ve	-ve	-ve	-ve	-ve	-ve	-ve	10	-ve	-ve
5	CoNcplx	14	11	10	15	12	9	14	12	9	15	13	9
6	CoScplx	15	13	9	14	12	9	13	11	7	13	11	9
7	NiNcplx	13	11	9	12	11	7	12	9	-ve	12	11	11
8	NiScplx	12	11	-ve	13	11	10	12	12	7	12	9	8
9	DMSO (Control)	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve

Table 5.0 Results of the Antifungal Activities of Ligands and their Metal (II) Complexes

S/No	Ligands and complexes	Diameter of Inhibition Zone (mm)											
		<i>Aspergillus flavus</i>			<i>Fusarium moniliforme</i>			<i>Rhizopus stolonifer</i>			<i>Fusarium solani</i>		
		Concentration (mg/ml)			Concentration (mg/ml)			Concentration (mg/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	½LSA	-ve	-ve	-ve	7	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
3	LNA	7	-ve	-ve	7	-ve	-ve	-ve	-ve	-ve	7	-ve	-ve
4	LSA	7	-ve	-ve	7	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve
5	CoNcplx	14	10	7	12	9	7	10	9	-ve	11	9	-ve
6	CoScplx	12	9	7	12	9	-ve	14	11	9	10	7	-ve
7	NiNcplx	9	7	-ve	10	9	-ve	10	9	-ve	8	-ve	-ve
8	NiScplx	13	8	-ve	9	9	-ve	10	10	-ve	9	-ve	-ve
9	DMSO (Control)	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve	-ve

3.5 Microbial Analysis

Cobalt and Nickel are among the bio essential metals in living organism and show antifungal and antibacterial properties against many pathogenic fungi and bacteria [16, 35, 36]. The results (tables 5.0 and 6.0) show that the metal complexes have greater antimicrobial activity than the ligands as revealed from the inhibition zones of the selected fungi and the bacteria [29]. The result of antifungal activity (table 6.0) revealed that the CoScplx show greater efficacy overall with (100 mm inhibition zone) followed by CoNcplx (98 mm), then NiScplx (68 mm) and finally NiNcplx (62 mm). Also, going by the total inhibition zone for antibacterial activity (table 5.0), CoNcplx show greater efficacy with 143 mm followed by CoScplx (136 mm), then NiNcplx (118 mm) and finally NiScplx 117 mm. The DMSO was used as control for both the antibacterial and antifungal analysis.

III. Conclusion

The synthesized compounds of Cobalt(II) and Nickel(II) complexes of 2-aminoaniline and 2-thiosylaniline show typical coloration, stable and unhygroscopic with favorable percentage yield. The FT-IR results guided in the determination of the synthetic route of the reaction and bonding processes. The UV-VIS of the ligands and complexes demonstrated characteristic behavior in the number of peaks splitted within the visible region (380-800 nm). The mass spectroscopy of the compounds confirmed the proposed structures through their m/z values according to their molecular weight. The synthesized complexes were found to be very effective in their antimicrobial activities when compared to the ligands especially CoScplx (antifungal) and CoNcplx (antibacterial), hence can be incorporated into antibiotic drugs.

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