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Synthesis, Characterization and Antimicrobial Activities of Schiff Base Derived From Glyoximehydrazine and Substituted Furan and Their Metal (II) Complexes

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ABSTRACT: A hydrazone Schiff base has been synthesized by the interaction of ethanolic solutions of antiglyoxime hydrazine with 2-acetyl-5-methylfuran to obtain the Schiff base L′. The complexes of Co(II), Ni(II) and Cu(II) of this Schiff base was synthesized and studied. The Schiff base and its metal(II) complexes were characterized based on solubility, percentage yield, elemental analysis, melting point/decomposition temperatures, infra- red spectral (FT-IR), conductivity measurement, magnetic susceptibility and water of crystallisation. Based on the results the prepared Schiff base and synthesized complexes are relatively soluble in most organic solvents but insoluble in distilled water. The melting point temperature of the ligand was 118^{o}C while the decomposition temperature for metal complexes was found to be between 295 -320^{o}C. The molar magnetic susceptibility of the complexes were in the range of 1.91 x 10^{-3} – 1.46 x 10^{-2}erg.G^{-2}mol^{-1}. The molar conductance values of 2.00 – 3.00ohm^{-1}cm^{2}mol^{-1} indicating non electrolytic nature of the complexes. The spectral data of the Schiff base showed band at 1641cm^{-1}, revealing the formation of the azomethine group in the Schiff base. A band between 776 and 791cm^{-1} indicate the metal nitrogen bond. The elemental analysis determination of the complexes and the Schiff base showed the metal ligand ratio of 1:2 (M:2L). The antibacterial and antifungal activities of the Schiff base and the metal(II) complexes were evaluated using disc diffusion method. The antibacterial assay was carried out on three pathogenic bacteria, *Salmonella typhimurium*, *Proteus aureginosa* and *Staphylococcus aureus*, and *two fungi viz: Candida utilis* and *Saccharomyces cerevisiae*. The Schiff base and the metal(II) compounds showed some antibacterial and antifungal activity.

Keywords: Metal(II) Complexes, Schiff base hydrazone, Antibacterial and Antifungal studies.

RESEARCH PAPER

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1.0 INTRODUCTION

Schiff bases are condensation products of primary amines with aldehydes or ketones they were first reported by Hugo Schiff in 1864. The most common structural feature of these compounds is the azomethine group (C=N) with the general formula RHC=N-R' where R and R' are alkyl, aryl, cyclo or heterocyclic groups which may be variously substituted, these compounds are also known as anils, imines, or azomethines [1, 2].

Schiff base metal complexes have been widely studied because they have industrial, antibacterial, antifungal, antiviral, anticancer, anticonvulsant and herbicidal applications [3]. Some complexes containing nitrogen and oxygen donor atoms in their structures are effective as stereospecific catalysts for oxidation, reduction, hydrolysis, biological activity and other transformation of organic and inorganic chemistry [4]. Schiff base metal complexes exhibit a large number of biological activities which include antibacterial, antifungal, antimalarial, antiproliferative, antiinflamatory, antiviral and antipyretic activities, they also used in agricultural and industrial applications [5]. Apart from their biological effects Schiff base metal complexes are also used in many areas such as fungicidal, dyes, pigments, polymer inhibitors, agrochemical, ion exchange, catalysis, electrical conductivity, nonlinear optics, magnetism and analytical chemistry [6].

Hydrazones are also a class of Schiff base containing C=NNH$_2$, as their functional group, they are formed by the condensation of substituted hydrazine with aldehyde or ketone [7]. Therefore they are related to aldehydes or ketones by the replacement of the oxygen by NNH$_2$ functional group.

Hydrazone Schiff bases contain two connected nitrogen atoms of different nature and a C=N double bond that in conjugated with alone electron pair of the terminal nitrogen atom. These structural fragments are principally responsible for the physical and chemical properties of hydrazone Schiff bases [8].

Hydrazones also constitute an important class of biologically active drugs molecules which has attracted attention of medical chemists due to their antimicrobial, antihypertensive, analgesic, anti-inflammatory, anti-tuberculosis, antiproliferative and antimalarial activities. They are formed usually by the action of hydrazine on ketones or aldehydes, hydrazone ligands and their metal complexes have important applications as anticancer, antimalarial, antioxidant [9].

Hydrazone is a versatile moiety that exhibits a wide variety of biological activities. Acyl hydrazones are very old class of molecules: the first example of N-acylhydrazines was mentioned in 1850 and a number of N-unsubstituted, and mono-disubstituted acylhydrazines were discovered and explored by the scientists worldwide [10, 11].

Hydrazone nucleus exhibited immense pharmacological activities. Hydrazones are present in many of the bioactive heterocyclic compounds that are of very important use because of their various biological and clinical applications. Hydrazone-based coupling methods are used in medical biotechnology as anticonvulsant, antidepressant, analgesic, anti-inflammatory, antiplatelet, antimalarial, antimicrobial, anticancer, vasodilator, antiviral, anti-HIV, antidiabetic and trypanocidal activities [12].

Recently a lot of work has been carried out in the literature on substituted furan but no one has reported the antimicrobial studies of the substituted furan with glyoxime hydrazone hence the interest.

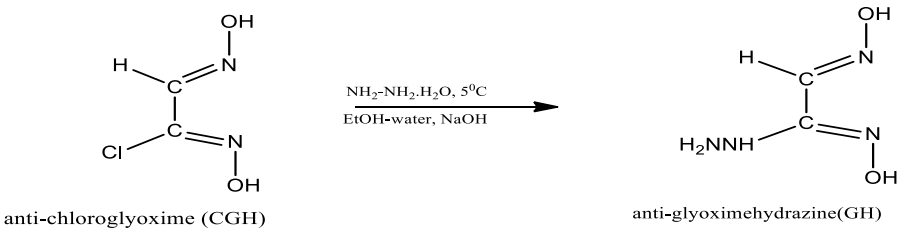
2.0 MATERIALS AND METHODS

2.1 Chemicals, Reagents and Apparatus

All glass wares used in this work were properly washed with detergent, rinsed several times with tap water. They were then soaked in a concentrated solution of nitric acid for about 2 – 3 hours after which they were rinsed 3 – 4 times with distilled water and dried in an oven maintained at 110^{o}C. All reagents and solvents used were of Analar grade and were used as supplied without further purification. All weighing were carried out on College B154 Metler Toledo electric balance. Melting point and decomposition temperatures were carried out on Stuart SMP 10 melting point apparatus. Determination of water of hydration was done on drying oven model DHO – 9053A. FT-IR spectra measurements were recorded using Agilent Technologies FT – IR spectrophotometer Carry 630, in the region of 400 – 4000cm^{-1}. The metal content of each complex was determined using Atomic Absorption Spectrophotometer (AAS), Bulk Scientific VGP 210. The elemental analysis (CHNS) was carried out at OEA labs., Callington, United Kingdom using a CE instruments (thermo) EA1110 Elemental analyser. The electrical conductivity measurements were also carried out using conductivity meter DDS-307, Jenway. The magnetic susceptibility measurements were carried out using magnetic measurement balance Sherwood scientific MK 01 model at room temperature. Three bacterial isolates were used in the test of microbial activity viz: *Salmonella typhimurium Proteus aureginosa* and *Staphylococcus aureus,* and two fungal isolates *viz: Candida utilis and Saccharomyces cerevisiae.*

2.2 Synthesis of intermediate Glyoximehydrazine

A solution of NaOH (0.4g, 10mmol) in 1cm^3 of distilled water was mixed with 10cm^3 of ethanol and 0.6cm^3 of hydrazinium hydroxide and cooled to 5^{o}C. A solution of chloroglyoxime (1.225g, 10mmol) in 5cm^3 of ethanol was added drop wise with stirring into the prepared mixture maintained at 5^{o}C. Stirring was continued for 15min at the same temperature to complete the reaction. The precipitate that formed was filtered, washed with cold ethanol and dried in a desiccator over phosphorus pentaoxide.



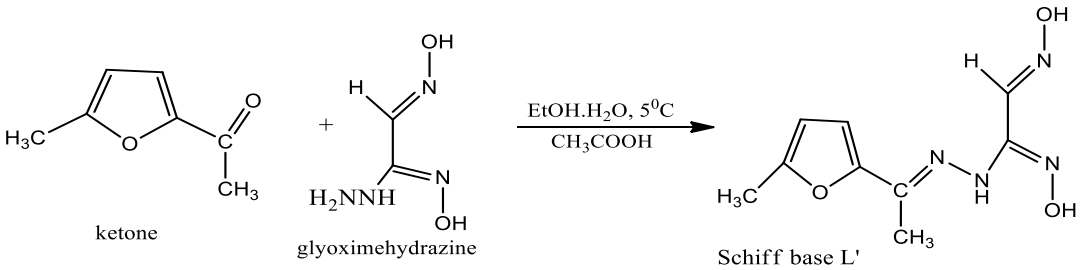
anti-chloroglyoxime (CGH) anti-glyoximehydrazine(GH)

Scheme 2.1: Preparation of glyoximehydrazine(GH)

2.3 Preparation of 2-acetyl-5-methylfuran glyoxime hydrazone (L′)

A cooled solution 5^0C of 2-acetyl-5-methylfuran (1mmol) in ethanol was added drop wise into a cooled solution 5^0C containing 1mmol of glyoximehydrazine and 3 to 5 drops of acetic acid with constant stirring. After the addition of the substituted furan was completed, the solution was stirred for 4 to 6 hours at room temperature. The resulting precipitate (L′), was separated, washed with water, ethanol and dried over phosphorus pentaoxide in a desiccator.

The equation for the reaction is presented below:

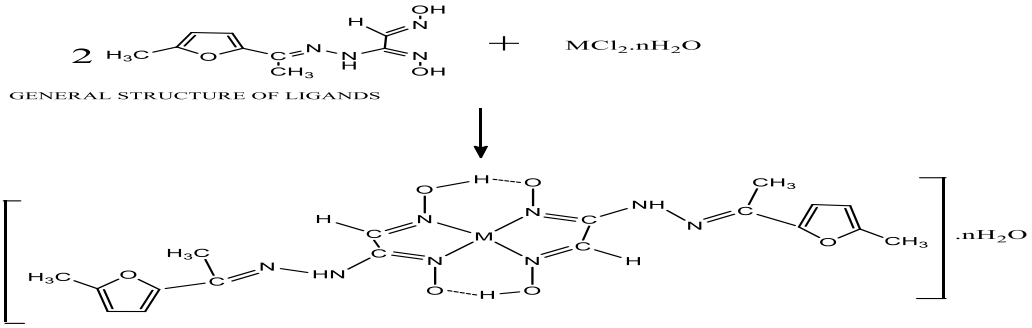


Scheme 2.2: Preparation of the Schiff base L'

2.4: Synthesis of Metal(II) Complexes

A solution of Co(II), Ni(II) and Cu(II) chlorides (1mmol) in $20cm^3$ of distilled water were added to 2mmol of Schiff base ligand (L′) in $15cm^3$ of ethanol with constant stirring. An initial sharp decrease of pH of the solution from 5.5 to 3.5 was observed. After raising the pH to 5.5 using aqueous NaOH solution, the reaction mixture was kept in a hot water bath (60^oC) for 2 to 3 hours to complete the precipitation. Then the precipitated complexes were filtered, washed with water and dried in a desiccator over phosphorus pentaoxide.

The general equation for the reaction is:
Where M = Co^{2+}, Ni^{2+} or Cu^{2+}



Scheme 2.3: Preparation of the metal(II) complexes

2.5 Antibacterial Activity Test

Antibacterial and antifungal activity of the synthesized Schiff base and their metal(II) complexes were tested using disc diffusion method on Mueller Hinton agar (MHA) and potato dextrose agar (PDA) sterile plates. Schiff bases and metal(II) complexes was dispensed into separately labelled wells using sterile 1mL insulin syringe. The preparations were incubated at 37^OC for 24 hours. Mean zones of inhibitions were measured using metre rule and recorded in millimetre [13].

Three bacterial isolates of which three viz: *Salmonella typhimurium Proteus aureginosa* and *Staphylococcus aureus*.

2.6: Antifungal Activity Test

Two fungal isolates *viz: Candida utilis and Saccharomyces cerevisiae*were were used to evaluate the antifungal activities of the Schiff base and its metal(II) complexes.

3.0: RESULTS AND DISCUSSION

Table 1: Physical propreties of the Schiff base and its metal(II) complexes

Compound	Molecular formula	Colour	Percentage yield (%)	Melting Temp.(^{o}C)	Decomp. Temp.(^{o}C)
L′	$C_9H_{12}N_4O_3$	Orange	65	118	-
$[CoL'_2].4H_2O$	$[Co(C_9H_{11}N_4O_3)_2].4H_2O$	Brown	61	-	311
$[NiL'_2].4H_2O$	$[Ni(C_9H_{11}N_4O_3)_2].4H_2O$	Green	65	-	308
$[CuL'_2].2H_2O$	$[Cu(C_9H_{11}N_4O_3)_2].2H_2O$	Green	60	-	300

Table 2: The Solubility Data of the Schiff base (L′) and its Metal(II) Complexes

SOLVENT COMPOUND	DistH_2O	MeOH	EtOH	Acetone	Hexane	$CHCl_3$	CCl_4	DMF	C_6H_6	DMSO
L′	IS	S	S	S	SS	S	SS	SS	SS	SS
$[CoL'_2].4H_2O$	IS	SS	S	S	S	SS	SS	S	SS	S
$[NiL'_2].4H_2O$	IS	SS	SS	S	S	SS	SS	S	SS	S
$[CuL'_2].2H_2O$	IS	SS	S	S	S	SS	SS	S	SS	S

Key: S = soluble, SS = slightly soluble and IS = Insoluble.
L′ = 2-acetyl-5-methylfuran glyoximehydrazone

Table 3: Molar Conductance Measurement for metal(II) complexes

Compound	Molecular Mass(g/mol)	Specific Conductance (ohm^{-1}cm^{-1})	Molar Conductance (ohm^{-1}cm^{2}mol^{-1})
$[CoL'_2].4H_2O$	577.4158	2.76 x 10^{-6}	2.76
$[NiL'_2].4H_2O$	577.1726	2.17x 10^{-6}	2.17
$[CuL'_2].2H_2O$	545.9982	2.43x 10^{-6}	2.43

L′ = 2-acetyl-5-methylfuran glyoximehydrazone

Table 4: Results for the Determination of the Metal Concentration in the Metal (II) Complexes

COMPOUND	Absorbance (y)	Concentration (x)(mg/l)	Metal percentage (%)
$[CoL'_2].4H_2O$	0.069	54.33	10.87
$[NiL'_2].4H_2O$	0.070	51.47	10.29
$[CuL'_2].2H_2O$	0.990	51.32	10.26

L′ = 2-acetyl-5-methylfuran glyoximehydrazone

Table 5: Magnetic Susceptibility Data for Metal(II) Complexes

Compound	Mass susceptibility Xg(erg.G^{-2}g^{-1})	Molar Susceptibility Xm(erg.G^{-2}mol^{-1})	Number Of unpaired Electron(s)	Geometry
$[CoL'_2].4H_2O$	1.40 x 10^{-5}	8.08 x 10^{-3}	3	Tetrahedral
$[NiL'_2].4H_2O$	6.16 x 10^{-6}	3.55 x 10^{-3}	2	Tetrahedral
$[CuL'_2].2H_2O$	3.85 x 10^{-6}	2.10 x 10^{-3}	1	Tetrahedral

L′ = 2-acetyl-5-methylfuran glyoximehydrazone

Table 6: IR Spectral Data (cm^{-1}) for the Schiff base (L′) and its Metal(II) Complexes

Compound	ν(N-H)	ν(O-H)	ν(C=N) oxime	ν(C=N) hydrazone	ν(C-H)	ν(M-N)
L′	3156	3085	1584	1640	2947-2918	-
$[CoL'_2]$ 4H2O	3551	3063	1681	1640	2973-2940	776
$[NiL'_2]$ 4H2O	3156	3115	1640	1640	2947-2918	791
$[CuL'_2]$2H2O	3353	3095	1640	1640	2947-2880	791

Table 7: Micro elemental Analysis Data for the Schiff base (L′) and its Metal(II) Complexes

Compound	Molecular Wt(g/mol)	Elemental %Found (Calculated)			
		C	H	N	M
L′	224.2188	48.67(48.21)	6.72(5.39)	22.43(24.99)	-
$[CoL'_2].4H_2O$	577.4158	39.03(37.44)	3.92(3.84)	19.74(19.41)	10.87(10.21)
$[NiL'_2].4H_2O$	**577.**1726	37.84(37.46)	3.93(3.84)	19.32(19.41)	10.29(10.17)
$[CuL'_2].2H_2O$	545.9982	37.10(39.60)	4.85(4.06)	20.40(20.52)	10.26(11.64)

Table 8: Antibacterial Sensitivity Test for the Schiff base (L′) and its Metal(II) Complexes

Compound	Concentration (µg/ml)	Bacterial Zone of Inhibition(mm)		
		Salmonella typhimurium	***Proteus Sp***	***Staphylococcus aureus***
L′	600	08	08	08
	300	06	06	06
	150	06	06	06
$[Co\mathbf{L'_2}].4H_2O$	600	08	06	07
	300	06	06	06
	150	06	06	06
$[Ni\mathbf{L'_2}].4H_2O$	600	10	08	06
	300	06	06	06
	150	06	06	06
$[Cu\mathbf{L'_2}].2H_2O$	600	11	10	08
	300	10	06	06
	150	06	06	06

L′ = 2-acetyl-5-methylfuran glyoximehydrazone

Table 9: Antifungal Sensitivity Test on the Schiff base (L′) and its Metal(II) complexes

Compound	Concentration(µg/ml)	Fungal zone of Inhibition (mm)	
		candida utilis	***saccharomyces cerevisiae***
L′	600	06	06
	300	06	06
	150	06	06
$[CoL'_2].4H_2O$	600	08	08
	300	06	06
	150	06	06
$[NiL'_2].4H_2O$	600	08	08
	300	06	06
	150	06	06
$[CuL'_2].2H_2O$	600	08	12
	300	06	10
	150	06	08

L′ = 2-acetyl-5-methylfuran glyoximehydrazone

DISCUSSION

The reaction of the prepared antichloroglyoxime with hydrazinium hydroxide in the presence of aqueous NaOH, produced antiglyoximehydrazine(GH). The antiglyoximehydrazine was separately reacted with 2-acetyl-5-methylfuran to produce the corresponding Schiff base hydrazone ligand labelled L′.

The reaction between the Schiff base hydrazone in the molar ratio 2:1 and aqueous solution of Co(II), Ni(II) and Cu(II) chlorides in ethanol produced the respective metal(II) complexes. The percentage yield of the Schiff base hydrazone were calculated 65% also the percentage yield of the corresponding metal(II) complexes were also calculated as 61, 65 and 60% respectively. The melting point of the hydrazone Schiff base and decomposition temperatures of the metal(II) complex compounds recorded were (Table 1).

Solubility of all the Schiff base and their metal(II) complexes were also determined in some common organic solvents in which the Schiff base is soluble in methanol and ethanol but insoluble in DMSO. All the metal(II) complexes are insoluble in distilled water but soluble in DMSO, the results were shown in (Table 2).

Molar conductance measurements were carried out in 10^{-3}mol dm^{-3} DMSO at 25^{O}C (at room temperature), molar conductivity values of the complexes are given in Table 3. the molar conductance of the synthesized metal(II) complexes of Co(II), Ni(II) and Cu(II) were in the range of 2.76, 2.17 and 2.43 $ohm^{-1}cm^{2}mol^{-1}$ respectively, these low values were agrees with those obtained in the literature. The molar conductivity result that revealed nonelectrolytic nature of the complexes [14] (Table 3).

The metal concentration in the complex compounds were determined using atomic absorption spectrophotometer (AAS) which were used to calculate the percentage of the metal(II) ion in the complex and the result obtained agreed with the theoretical values, the values were recorded in (Table 4).

Table 5 showed the magnetic susceptibility results of the complexes taken under room temperature. The values for the magnetic moments for all the metal(II) complexes conformed to a tetrahedral geometry, the Schiff base ligands all have magnetic moments values suggesting high spin complexes.

The Schiff base (L′) was in accord with the previously reported hydrazones [15]). The IR spectra of the Schiff bases showed strong bands at 1581 - 1607cm^{-1} assigned to ν(C=N) stretching frequencies, suggesting the formation of Schiff base. This bands shift to higher frequency of 1607 – 1684cm^{-1} in the metal(II) complexes revealing that coordination has taken place between the metal(II) ions and the respective Schiff base through the (C=N) of the oxime portion [16] (Table 6).

Table 7 showed the results of micro elemental analysis for the elements (NCHS) in the Schiff base hydrazones and their corresponding Co(II), Ni(II) and Cu(II) complexes, the results obtained from elemental analysis were almost the same as the results calculated theoretically.

The antibacterial assay carried out on three *Salmonella typhimurium, Proteus auregonosa*., and *Staphylococcus aureus,* zones of inhibition based upon size around each of the discs were measured in millimetre and recorded. The ligands and metal(II) complexes affect the bacterial isolates differently; the ligands showed lower activity compared to the metal(II) complexes. Generally the anti-bacterial activity increases with increasing concentrations. For ligand L´ it showed high activity at higher concentration (Table 8).

The antifungal results of (L´) Schiff base hydrazone shown against the two fungal isolates indicate no activity at any concentrations with the zone of inhibition were only within the disc i:e 6mm. However the results of complex [CuL_2'].$2H_2O$ showed a higher activity of 10mm at 600µg/ml while complexes [CoL_2'].$4H_2O$ and [NiL_2'].$4H_2O$ showed lower activity of 8mm at the same concentration (Table 9).

CONCLUSION

The hydrazone Schiff base and their metal(II) complexes were successively prepared and analysed. The results obtained of percentage yield for the ligand is 65% and the metal(II) complexes range from 61, 65 and 60% respectively. The metal ligand ratio for all the metal(II) complexes from elemental analysis was found to be (1:2). The magnetic behavior of all the complex compounds have been determined and found to be paramagnetic all the metal(II) complexes tend to be tetrahedral. The relatively low molar conductance values indicated their non-electrolytic nature. The Infra-red spectral data of the ligands when compare to those of their respective metal(II) complexes showed that coordination of the metal ion to the ligand is via azomethine nitrogen and phenolic (OH) of the hydrazone Schiff base.

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