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Research Article

Synthesis, Characterization and Antimicrobial Activity of Mixed Ligand Metal (II) Complexes Derived from 2,2- Bipyridine and Oxalic Acid

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ABSTRACT
The synthesis of the metal (II) mixed ligand complexes of Mn, Co, Ni and Cu was carried out successfully by the reaction of the metal (II) chloride with the ligands 2,2-bipyridine and oxalic acid and characterized on the bases of solubility, decomposition temperatures, FTIR, UV-visible data, molar conductance, magnetic susceptibility, and elemental analysis. The analyses revealed that the metal complexes were soluble in DMSO, DMF and insoluble in certain organic solvents. It was discovered that the decomposition temperatures ranged from 243 - >300^{o}C. The high decomposition temperature shows that the complexes are stable in nature. The IR data show that the mixed ligands were coordinated to each of the metal (II) ions. The non-electrolytic nature of all the complexes is indicated by their low molar conductance values (4.72-29.77 ohm^{-1} cm^{2} mol^{-1}). Magnetic moment values (1.780-5.1085BM) of the complexes showed that all are paramagnetic in nature. The elemental analysis results of the ligands and the metal (II) complexes shows that the calculated percentages of C, H and N in the compounds and the experimental values(found) were in good agreement. This agrees with the proposed structures of the complexes. The antibacterial and antifungal activities of the ligands and metal (II) were carried out. The metal complexes exhibit moderate antibacterial and antifungal activities compared to the free ligands. Generally, the activity increases with increase in concentrations. But their activities are lower than that of the controls.

Keywords: 2,2- Bipyridine; Antimicrobial; Characterization; Ligand metal (II); Oxalic acid; Synthesis

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INTRODUCTION

Mixed-ligand metal complexes were found to be particularly useful because of their potential to bind DNA via multitude of interactions and to cleave the duplex by virtue of their intrinsic chemical, electrochemical and photochemical reactivity (Bayeh, 2019). Metal complexes play a significant role in various biological and chemical applications due to their unique structural and electronic properties. Many chelating ligands containing O, S, and N donor atoms and their metal complexes have been proven to exhibit distinct antimicrobial activities against many multidrug-resistant strains of bacteria and fungi (Kayode, *et al*., 2023).

Transition metals have an important place within medicinal inorganic chemistry. They exhibit different oxidation states and can interact with a number of negatively charged molecules. This activity of transition metals led to the recent development of drugs which are based on metals and are considered to be potential candidates for pharmacological and therapeutic application (Selvaganapathy and Raman, 2016).

Organic bidentade ligand such as oxalic acid or amino acids is an important class of ligand in coordination

chemistry and find extensive application in different fields such as in the field of medicines and bioinorganic chemistry (Taghreed and Ghassan, 2017).

2,2-bipyridine (Figure 1) is a chelating ligand, it has been used as bridging ligand so it found different application in coordination chemistry. It forms a 5membered chelate ring which is stable upon coordination of metal. Mixed ligand which contains 2,2bipyridine and other bidentate ligand are antineoplastic agents, these compounds exhibit cytotoxicity, antitumor effect and genotoxicity also have been bactericidal, bacteriostatic toward many gram-positive bacteria but they are in-effective against gram-negative organism (Hayder and Nesser, 2018).

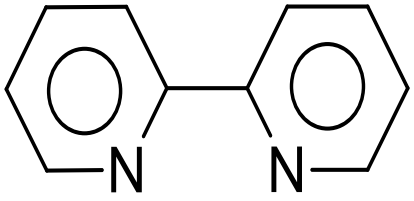


Figure 1: Structure Of 2,2-bipyridine

Oxalic Acid **(**ethanedioic acid) or called Wood bleach (Fig 2), acts as bidentate ligand in the form of the dianion, having two-carboxylate groups (and forms a 5 membered ring with the metal ion). The oxalate complexes of metal ions have been extensively studied, the sustained interest in their study in solution is a consequence of their use as model systems in the investigation of physio- chemical property (Taghreed and Ghassan, 2017).

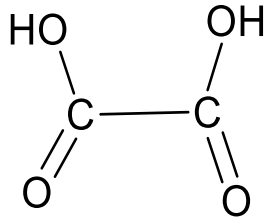


Figure 2: Structure Of Oxalic Acid

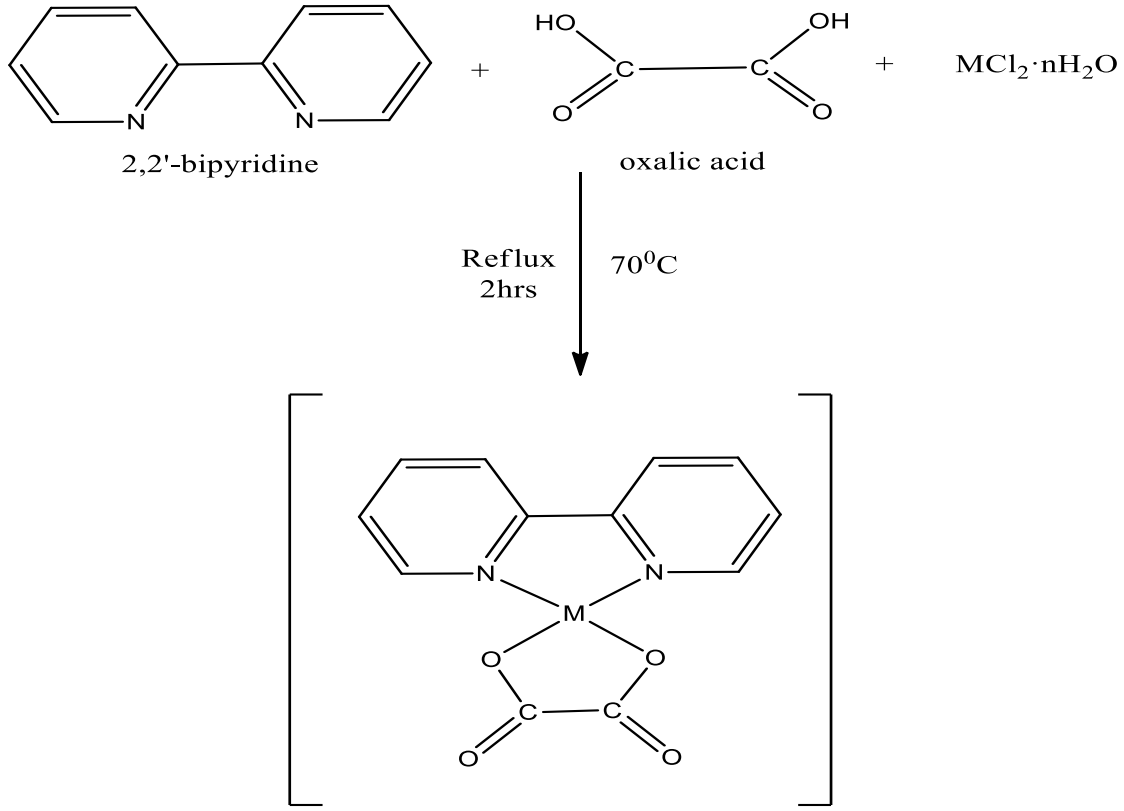
MATERIALS AND METHODS

All reagents and solvents used were of analytical grade and used without any further purification. The 2,2-bpyridine and oxalic acid used for the preparation of the mixed ligand complexes were obtained from Sigma Aldrich. The metal (II) chlorides were obtained from Sigma Aldrich. The solvents used: ethanol, methanol, acetone, dimethyl sulphoxide, dimethyl formamide, n-Hexane, chloroform, diethylether, were all obtained from Sigma Aldrich. Bacterial isolates **(***Staphylococcus aureus, Escherichia coli, Klebsiellae pneumonae*) and fungal isolates (C*andida albicans, Aspergillus niger, Tinea capitis*) were obtained and identified in Biology Laboratory, Department of Science Laboratory Technology. The melting point and decomposition temperature of ligands and complexes were recorded with Stuart SMP10 simple digital melting point apparatus. The molar conductance of the complex ions was determined using Jenwey 4010 conductivity meter. FT-IR spectra were recorded on Agilent Technologies spectrophotometer (Cary 630). The magnetic susceptibility measurement was carried out using a Sherwood Scientific Magnetic Susceptibility Balance at Chemistry Laboratory, Department of Science Laboratory Technology, Federal Polytechnic Daura, Katsina State. The UV spectra were collected on Perkin Elmer Lamda- 35 UV – spectrophotometer and elemental analysis was conducted at Al-Azhar University, Fermentation Biotechnology & Applied Microbiology (Ferm-BAM) Centre, Cairo, Egypt.

Synthesis of Mixed Ligands Complexes

To an ethanolic solution containing [$CuCl_2.2H_2O$] (0.170g, 1mmol), an ethanolic solution of 2,2-bipyridine (0.156g, 1 mmol) and ethanolic solution of Oxalic acid (0.09g, 1mmol) were added drop wise with constant stirring. After complete addition the reaction mixture was refluxed for 2hours and cooled to room temperature. The resulting precipitate was filtered and dried in vacuum desiccator over anhydrous $CaCl_2$ (Saifun *et al*., 2020).

Similar method was used in preparation of other metal (II) complexes (Scheme 1).



Scheme 1: Preparation of mixed ligand metal (II) complexes
Metal (II) Complex; M= Mn, Co, Ni and Cu

RESULTS AND DISCUSSION

The results obtained from the synthesis, characterization and antimicrobial activity of the mixed ligands and synthesized metal complexes are shown in the tables below.

The complexes synthesized were colored ranging from, emerald green for nickel (II) complex, turquoise blue for copper (II) complex, cream for manganese (II) complex and pink for cobalt (II)complex. The percentage yield of the complexes ranges from 75% to 98%, with Cu (II) complex recording the highest yield of 98% while Co (II) complex recorded the lowest yield with 75% as presented in table 1. The complexes decomposed at a temperature ranging from 243 - 341^{o}C (Table 1). The high decomposition temperature shows that the complexes are stable in nature.

The ligands and their complexes were further characterized by carrying out solubility test to determine their nature in different organic solvents such as ethanol, methanol, acetone, DMSO, chloroform, DMF, n-Hexane, diethyl ether, CCl_4 and pet ether. The results obtained were presented in table 2. The ligands and complexes are completely soluble in DMSO and DMF and were slightly soluble in ethanol and methanol. The ligands and complexes were found to be insoluble in chloroform, diethyl ether, CCl_4, pet ether, acetone, and n-hexane.

The molar conductance has proven to be a very crucial tool used in the investigation of the electrolytic nature of inorganic compounds. The molar conductivity of synthesized complexes in DMSO solution (10^{-3}) as listed in table 3 were in the range 4.72-29.77 $Ohm^{-1}\ cm^2\ mol^{-1}$ indicating the non-electrolytic character of the complexes.

Table 4, shows the IR spectra data of the compounds 2,2-bipyridine, oxalic acid and the metal (II) complexes respectively. Characteristic bands of carboxylate groups in the usual region of 1405-1399 cm^{-1} region for symmetric stretching are observed. Their positions and intensities are similar to those reported for other oxalic acid and their complexes (Hayder and Nesser 2018). The complexes display band in the region of 1659-1614 cm^{-1} and 1328-1294 cm^{-1} due to ϑ(C=O) and ϑ(C-O) respectively,

significantly lower than that of free ligand. The C-N stretching is at 1454-1447 cm^{-1}. The in-plane and out-plane ring deformation modes of heterocyclic amines were observed at 641 cm^{-1} and 608 cm^{-1} respectively undergoing a positive shift in mixed ligand complexes confirming their coordination through nitrogen. The presence of metal nitrogen bonding in the complexes is confirmed from the appearance of ϑ (M-N) modes at 622-474 cm^{-1} in the spectra of the complexes and ϑ(M-O) appearance at 641-608 cm^{-1}.

The values of the effective magnetic moment of the metal (II) complexes were summarized in table 5. The effective magnetic moment(μeff) values obtained were 1.993BM, 1.780BM, 5.1085BM, and 4.612BM for Ni (II), Cu (II), Mn (II) and Co (II) complexes respectively were all within the range reported for tetrahedral geometry around the respective metal ions (Saifun *et al*., 2020).

UV- spectra of all the complexes were recorded in DMSO at room temperature (Table 6). The absorption wavelength for the ligand 2,2-bipyridine exhibit bands at 244nm and for the complexes ranging from 234-262nm attributed to π - π * and transition within the ligand oxalic acid exhibit band at 250nm and for the complexes ranging from 232-248nm attributed to π - π* transition. Other absorption wave length in the range of 294-275nm assigned to n- π * transition.

The elemental analysis results of the ligands and the metal (II) complexes are presented in table 7. The results of the calculated percentages of C, H and N in the compounds and the experimental values (found) with slight differences. This agrees with the proposed structures of the complexes as shown in Fig 2.

Anti-fungal studies were carried out using well diffusion technique on Potato Dextrose Agar (PDA) against *Candida albicans, Aspergillus aiger and Tinea capitis* as shown in Table 8. The data revealed that the metal (II) complexes showed moderate activity than the ligands. It was further observed that an increase in concentration increases the activity of the compounds against the three fungal isolates. The Cu (II) andNi (II) complex shows less activity against *Tinea capitis* followed by Co (II) and Mn (II)complex. The Co (II) complex shows moderate activity against *Aspergillus niger*. The Mn (II) complex exhibited less activity against *Candida albican* they all exhibit less activities when compared with the standard drug Nystatin (500μg/ml).

The *in vitro* anti-bacterial activity of the ligands and its respective metal (II) complexes was carried out against three bacterial isolates, (*Staphylococcus aureus, Escherichia coli* and *Klebsiellae pneumonae*) using Muller-Hinton Agar. Well diffusion method with DMSO as solvent was used. The results indicated that the complexes exhibited moderate anti-bacterial activity against the ligand which generally increase with increase in concentration. The complexes showed more potency in inhibiting the growth of microorganisms than the ligands under similar experimental conditions which might be due to chelation. The data obtained from table 9 indicated that Co (II) complex exhibited higher activity against *Klebsiellae pneumonia*, followed by Cu (II) complex with moderate activity against *Escherichia coli* while Ni (II) complex showed low activity against *Staphylococcus aureus* theiractivities were all low in comparism with standard drug Amoxicillin (500μg/ml) (Hossain *et al*., 2019).

Based on the analytical, spectroscopic studies and literature the structure of the complex can be propose as Figure 3.

Table 1: Some Physical Properties of the Ligand and its corresponding metal (II) Complexes

Compound	Colour	Melting point(°C)	Decomposition Temp (°C)	% Yield
BP	White	75	-	-
OA	White	190	-	-
[Mn (OA)(BP)]	Cream	-	339	92
[Co (OA)(BP)]	Pink	-	326	75
[Ni (OA)(BP)]	Emerald Green	-	341	97
[Cu (OA)(BP)]	Turquoise blue	-	243	98

where BP =2,2- Bipyridine, OA = Oxalic Acid, D. Temp.= Decomposition Temperature.

Table 2: Solubility test of ligands, Mn (II), Co (II), Ni (II) and Cu (II) complexes.

Solvents	BP	OA	[Mn (OA)(BP)]	[Co (OA)(BP)]	[Ni (OA)(BP)]	[Cu (OA)(BP)]
Ethanol	SS	S	SS	SS	SS	SS
Acetone	IS	SS	IS	IS	IS	IS
Methanol	SS	S	SS	SS	SS	SS
Chloroform	IS	IS	IS	IS	IS	IS
CCl_4	IS	IS	IS	IS	IS	IS
DMF	S	S	S	S	S	S
DMSO	S	S	S	S	S	S
Pet Ether	IS	IS	IS	IS	IS	IS
Diethylether	IS	IS	IS	IS	IS	IS
n-Hexane	IS	IS	IS	IS	IS	IS

Where, BP = 2,2-Bipyridine, OA = Oxalic Acid, DMSO = Dimethylsulfoxide, DMF = Dimethylformamide, CCl_4 = Carbontetrachloride, S = Soluble, SS = Slightly Soluble, IS = Insoluble.

Table 3: Molar conductance of Metal (II) complexes in 10^{-3}M DMSO solution

Compounds	Electrical Conductance ($Ohm^{-1} cm^{-1}$)	Molar Conductivity ($Ohm^{-1}cm^2 mol^{-1}$)
[Mn (OA)(BP)]	29.77×10^{-6}	29.77
[Co (OA)(BP)]	6.43×10^{-6}	6.43
[Ni (OA)(BP)]	4.72×10^{-6}	4.72
[Cu (OA)(BP)]	7.33×10^{-6}	7.33

Where BP = 2,2-Bipyridine, OA = Oxalic Acid

Table 4: IR spectra of the Ligands and Mn (II), Co (II), Ni (II) and Cu (II) complexes

Compounds	V(C=N) cm^{-1}	V(M-N) cm^{-1}	V(M-O) cm^{-1}	V(C-O) cm^{-1}
BP	1454	622	-	-
OA	-	-	-	1328
[Mn (OA)(BP)]	1443	470	630	1316
[Co (OA)(BP)]	1447	474	619	1316
[Ni (OA)(BP)]	1447	474	608	1316
[Cu (OA)(BP)]	1447	470	641	1294

Where BP = 2,2-Bipyridine, OA = Oxalic Acid.

Table 5: Magnetic susceptibility values of the metal (II) complexes

Compound	Mass Susceptibility, Xg($gmol^{-1}$)	Molar Susceptibility, Xm($gmol^{-1}$	μeff (B.M)	Magnetic Property
[Mn(OA)(BP)]	3.660×10^{-5}	1.095×10^{-2}	5.1085	Paramagnetic
[Co(OA)(BP)]	2.944×10^{-5}	8.924×10^{-3}	4.612	Paramagnetic
[Ni(OA)(BP)]	5.502×10^{-6}	1.667×10^{-3}	2.030	Paramagnetic
[Cu(OA)(BP)]	3.872×10^{-6}	1.330×10^{-3}	1.780	Paramagnetic

where BP = 2,2-Bipyridine, OA = Oxalic Acid.

Table 6: UV Visible Spectral data of ligands and metal complexes

Compound	$C=C_{aro}$ (π-π*) nm	C=O (π-π*) nm	C=N (π-π*) nm	C=O (n- π*) nm	C=N (n- π*) nm
OA	-	250	-	294	-
BP	228	-	244	-	298
[Mn (OA)(BP)]	226	232	234	274	306
[Co (OA)(BP)]	208	244	254	275	312
[Ni (OA)(BP)]	210	248	260	272	310
[Cu (OA)(BP)]	224	234	262	273	302

where BP = 2,2-Bipyridine, OA = Oxalic Acid.

Table 7: Micro-analytical Data of the Ligands and their Metal Complexes.

	Percentage of Elements Observed (Calculated)		
Compounds	**C**	**H**	**N**
OA	21.30 (21.68)	8.60(7.61)	--
BP	73.20(74.90)	6.12(5.16)	17.40 (17.94)
[Mn (OA)(BP)]	43.20 (43.50)	2.58 (2.70)	1.23 (2.34)
[Co (OA)(BP)]	43.80 (44.55)	4.70 (4.66)	5.80 (6.24)
[Ni (OA)(BP)]	58.40 (57.58)	1.66 (2.66)	2.34 (2.25)
[Cu (OA)(BP)]	56.20 (55.92)	2.23 (3.52)	3.44 (3.15)

Where BP = 2,2-Bipyridine, OA = Oxalic Acid.

Table 8: Antimicrobial Sensitivity test of Complexes and Ligand against Fungal isolates using agar well- diffusion Method Diameter of zone of inhibitions (mm)

Compound	*Candida albicans*			*Aspergillus niger*			*Tinea capitis*		
	1000 (µg/ml)	500 (µg/ml)	250 (µg/ml)	1000 (µg/ml)	500 (µg/ml)	250 (µg/ml)	1000 (µg/ml)	500 (µg/ml)	250 (µg/ml)
OA	18	17	15	16	14	12	19	17	14
BP	23	20	17	17	15	12	16	14	13
[Mn (OA)(BP)]	16	14	12	13	12	9	12	10	06
[Co (OA)(BP)]	12	11	8	15	13	11	14	12	10
[Ni (OA)(BP)]	16	14	13	13	12	10	15	13	11
[Cu OA) (BP)]	14	12	10	13	11	8	15	13	11
Nystatin(500µg/ml)		35			33			28	

Where BP = 2,2-Bipyridine, OA = Oxalic Acid.

Table 9: Antimicrobial Sensitivity test of Complexes and Ligand against Bacterial isolates using agar well- diffusion Method Diameter of zone of inhibitions (mm)

Compound	*Staphylococcus aureus*			*Escherichia coli*			*Klebsiellae pneumonia*		
	1000 (µg/ml)	500 (µg/ml)	250(µg/ml)	1000 (µg/ml)	500 (µg/ml)	250 (µg/ml)	1000 (µg/ml)	500 (µg/ml)	250 (µg/ml)
OA	18	16	14	17	15	13	16	14	12
BP	22	18	15	19	17	13	15	13	11
[Mn (OA)(BP)]	15	13	12	14	12	10	12	9	06
[Co (OA)(BP)]	19	17	15	16	14	12	15	13	11
[Ni (OA)(BP)]	13	12	10	15	13	12	14	12	10
[Cu(OA)(BP)]	17	15	13	18	16	14	12	11	8
Amoxicillin(500µg/ml)		40			35			32	

Where BP = 2,2-Bipyridine; OA = Oxalic Acid

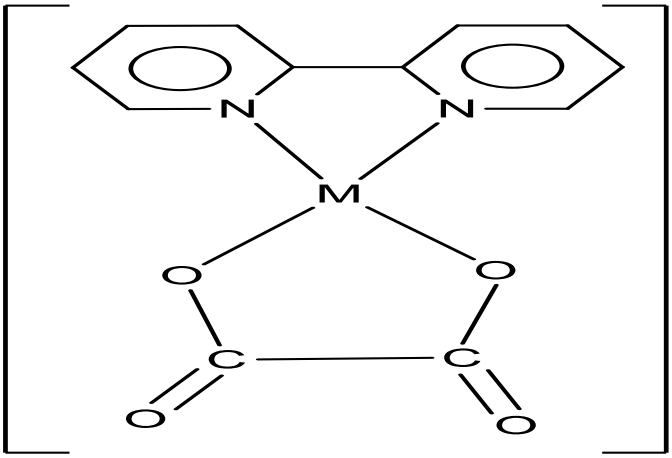


Figure 3: Proposed structure of the complex
M= Mn, Co, Ni and Cu

CONCLUSION

The mixed ligand and its metal complexes of Mn (II), Co (II), Ni (II) and Cu (II) were synthesized and characterized on the basis of spectroscopic data (FT-IR and UV-Visible) and analytical data (molar conductance, magnetic susceptibility measurements and elemental analysis). The complexes were found to be non-electrolytic with tetrahedral geometry having variable degree of solubility in common organic solvents. The IR and elemental analysis method data agreed with the proposed structure of the metal (II) complexes. The prepared complexes also show promising antimicrobial activity.

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