SYNTHESIS AND CHARACTERISTIZATION OF SOME METAL COMPLEXES OF CITRIC ACID MONOHYDRATE

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ND/23/SLT/PT/0737

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ILORIN, KWARA STATE, NIGERIA.

IN PARTIAL FULFILLMENT OF THE REQUIREMENT FOR THE AWARD OF NATIONAL DIPLOMA (ND)
IN SCIENCE LABORATORY TECHNOLOGY

JULY, 2025.

CERTIFICATION

This is to certify that this project work was carried out by **JAMIU OLUWATOYIN OMOLEWA** with matriculation number **ND/23/SLT/PT/0737** and has been read and approved as meeting the requirements for the award of National Diploma (ND) in Science Laboratory Technology (SLT), Institute of Applied Sciences (IAS), Kwara State Polytechnic, Ilorin, Kwara State.

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DEDICATION

This project is dedicated to Almighty God for his merciful and blessing throughout the completion of this programme, Despite all the odd, And who blessed me with good health all through my stay in the school.

Also to our dearest parents Mr and Mrs JAMIU who all has supported us in all ways especially their prayers over our life and siblings, You are all source of my inspiration.

ACKNOWLEDGEMENT

When God says yes, who can say no? All glory, adorations go to Almighty God, the creator of the whole universe for making it possible for me to excel in my academic pursuit.

I am indebted to my project supervisor; in the person of **Mr Muritala Olanrewaju .O.** for his invaluable contribution and suggestion towards making this project a successful one, may Almighty God continue to enrich you with knowledge and your family sir. I also extend my gratitude to the **HOD Mr Abdulkarem** Usman and lecturers in the Department of Science Laboratory Technology for the knowledge impacted on me. I say thank you sir/ma.

My profound gratitude and appreciation goes to my lovely family and loved ones **Mr and Mrs JAMIU, Alabere** for their immense and unquantifiable effort financially, morally, spiritually, materially and parental care, to make my education career a success and reality.

Special thanks to all those who provided me with necessary literature for this work and to all those I didn't mention here I am thankful to you all as well.

ABSTRACT

Citric acid metal complexes were prepared on refluxing using appropriate amount of metal salt to ligand in the mole ratio 1:2

The complexes were characterized using melting point, solubility test, infrared and ultraviolent-visible spectroscopy. Octahedral geometry were proposed for the complexes due to the coordination of the metal to the oxygen atom of the citric acid monohydrate.

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CHAPTER ONE

1.0 INTRODUCTION

Citric acid metal complex is the study of interaction between the ligand and the metal. Citric acid as the Lewis base that is electron donor have lone pair electrons which can be donated to a metal for coordination.

The studies of electron pair donated from ligands to metals and other acceptors form a coordination link. The approach in bonding complex was extended by Pauling and developed into the valence bond theory of metal-ligand bonding.

In this project, complexes were derived from the interaction between citric acid and metal complexes. Hence, the word CITRIC ACID METAL COMPLEX.

LIGANDS

A ligand is in an atom, ion or molecule that bond to the metal which involve donation of one or more of the lone pair electrons.

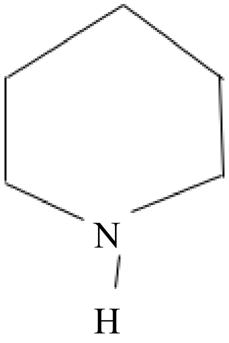
Ligands are viewed to be a Lewis base serving as an electron donor, in other words, ligands in a complex dictate the reactivity of the central atom.

$$\text{Lewis acid + Lewis base} \longrightarrow \text{Coordination Compound}$$

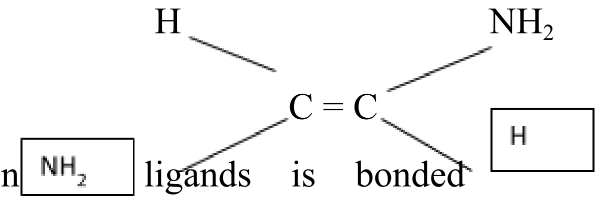
In coordination chemistry, ligands in metal complexes that are directly bonded to the metal is known to be the inner sphere" ligands, while ligands that are not directly attached to the metal but are bonded weakly is called the outer sphere ligands". The complex of the metal with the inner sphere ligands is called coordination complex which can be neutral, cationic or anionic and the complex along with its counter ions is called a coordination compound.

Many ligands are capable of binding metal ion through multiple sites usually because they have lone pairs on more than one atom and ligands that bind via more than one atom are termed chelates"

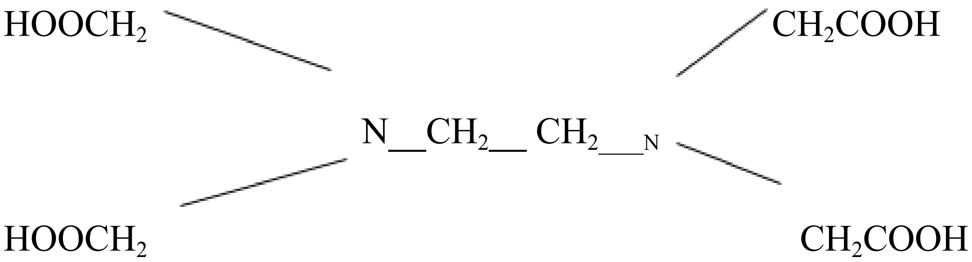
An example of ligand that binds through one site known as monodentate.am example is Piperidine



Two sites as bidentate such as Ethylene diamine{en}



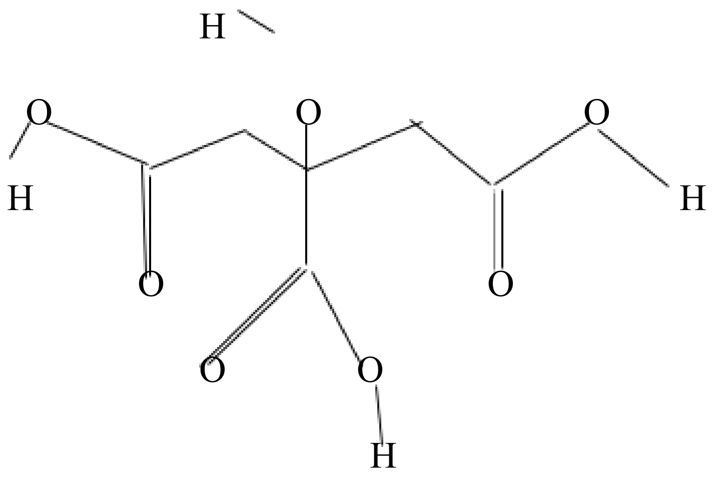
When ligands is bonded through three or more sites, is known to be polydentate”. An example is the chelating agent Ethylenediamine teteracetic acid (EDTA) which is able to bond through six sites



1.1 CITRIC ACID

Citric acid is a weak organic acid found in citric fruits. It is a good, natural preservative and is also used to add an acidic (sour) taste to food and soft drinks. In biochemistry, it is important as an intermediate in the citric acid cycle and there occurs in the metabolism of almost all living things. It also serves as an environment cleaning agent and act as antioxidant and a lubricant. [1]

Citric acid exists in a variety of fruits and vegetables, but it is most concentrated in lemons and limes, where it can comprise as must as 8% 9of the dry weight of the fruit.



Citric acid chemicals formula: $C_6H_8O_7$. Its structure is reflected in its IUPAC name 2-hyroxypropane -1,2,3-tricarboxylic acid.

1.1.1 HISTROY OF CITRIC ACID

The discovery of citric acid has been credited to the 8th century Iranian chemist Jabri Ibn Hayyan (Geber) {2,3,4}.Medieval scholars in Europe were aware of the acidic nature of lemon and lime juices such knowledge is recorded in the 13th century encyclopedia speculum Majus (The Great Mirror), compiled by Vincent of Beauvais. Citric acid was first isolated in 1784 by the Swedish chemist Cash Wilhelm Scheele, who crystallized it from lemon juice {5}{6}. Industrial scale citric acid production began in 1860, based on the Italian citric fruit industry.

In 1893, C. Wehmer discovered the Penicillium mold could produce citric from sugar. However, microbial production of citric acid did not become industrial important until World War I disrupted Italian citric exports. In 1917, the American food chemist James Curie discovered that certain strains of the mold Aspergillius Niger could be efficient citric producer and Pfizer began industrial –level production using this technique two year later following by citrique Belge in 1929.

1.1.2 PRODUCTION OF CITRIC ACID

In this production technique which is still the major industrial route to citric acid used today cultures of Aspergillus Niger are fed on a Sucrose or glucose containing medium to produce citric acid. The source of sugar is corn steep ligour,{7} molasses hydrolyzed corn starch or other inexpensive sugary solutions {8}. After the mold is filtered out of the resulting solution, citric acid is isolated by precipitating it with lime (calcium hydroxide) to yield calcium citric salt, from which acid is rdgenerated by treatment with sulphuric acid.[9]

1.2 METALS

A metal is an element that readily loses electron to form positive ions (cautions) and has metallic bond between metal atoms. Metals form ionic bond with non-metals. They can also be described as a lattice of positive ions surrounded by a clouding of delocalized electrons.

Properties of Metals

- They are good conductor of heat and electricity
- They are ductile. Lustrous and malleable

- They are sold at room temperature
- Most metals have high melting and boiling point while some have low melting and low boiling point.
- They are sonorous that is, they give a note when hitted

1.2.1 COPPER

Copper is one of the less reactive metals and belongs to the noble metal earlier discussed which include silver and gold. It is very stable in air and can be found in the free metallic form in few places. Its symbol Cu is derived from the Latin name cuprum". Its major ores include Chalcopyrite (CuFeS), Cuprite (Cu_2O) e.t.c which are used in the extraction of copper.

Physical Characteristics of Copper

- It is a soft, red solid with lustre
- It has a relative density of 8.95
- It is malleable and ductile
- Its melting and boiling points are 1462 and 2562 respectively.
- It is good conductor of heat and electricity
- It has atomic weight of 63.54gmol and atomic number of 29.

Copper has a {Ar} $3d^{10}4s^1$ configuration which exhibits various oxidation state of +I, +2, +3 and +4. The most common structure formed by copper complexes are square planar and octahedral geometry which exhibits a coordination number of +4. For example, $\{Cu(NH_3)_4\}^{2+}$ has octahedral geometry

Uses of Copper

COMPUTERS

Copper is vital for computer to work. Copper is used in buildimg the intedreated circuits, chips and the printed circuit boards of the computer alone. Copper is becoming more and more popular to use in the layers of the build-up of a chip. IBM announced a plan to use copper in its computer chip rather than aluminum. Doing so would make the computer to be cheaper and would allow it to make faster calculations.

ELECTRICITY

Copper is often used as a conductor of electricity. Almost all electricity devices rely on copper wiring because copper is inexpensive and highly conductive. The conductivity of copper is second to only silver. The reason why copper is a good conductor is because there are a lot of free electrons that can carry the flowing current efficiently. These free electrons do not remain permanently associated with the copper atoms, instead they form an electron cloud around the outside of the atom and are free to move through the solid quickly.

CURRENCY

Copper is often used in currency. From 1909 to 1982, the American penny was 95% copper. The other 5%was either zinc or bronze depending on the year. The only exception was 1943. That year penny had zinc-plated steel in it. Unfortunately, the steel was magnetic and kept getting stuck in vending machines. Also, the zinc corroded easily and was therefore often mistake for a dime. In the early 1980s, copper increased in value. So in 1982, the United States switched the penny's core to zinc and coated it with copper.

HEALTH

Two radioactive isotopes of copper, copper-64 and copper-67, are used in medicine. The first, copper-64, is used to study brain function and to detect Wilson's diseases. Wilson's disease occurs when a person cannot eliminate copper from his or are body. Copper-67 treat cancer. When the isotope is injected into the body it goes to cells that are cancerous and gives off radiation that can kill the cancerous cells.

1.2.2 LEAD

Lead is derived from the Latin word plumbum" and thus has the symbol Pb. It occurs combined in nature and its major ore is galena(Pbs) which contains 86.6% lead. Other include cerussite ($PbCo_3$) and arylesite ($PbSO_4$)

Physical properties of Pb

- It is a bluish grey solid with characteristic lustre
- It has a relative density of 11.34
- Its melting and boiling points are 1327.46^{o}C and 1749^{o}C
- It's also malleable, less ductile and a good conductor of heat and electricity.
- It forms amphoteric oxide.

lead has a {Xe} $4f^{14}$ $5d^{10}$ $6s^2$ $6p^2$ configuration which exhibits oxidation state of +2 and +4 complexes formed by Pb are mostly tetrahedral in geometry and have a coordination number of 2

1.3 COMPLEXES

A complex is used to describe molecules formed by the combination of ligands and metals ions. It also implies a reversible association of molecules, atom or ions through weak chemicals bonds and some metals complexes are formed virtually irreversibly and are bound together by bonds that are quite strong.

Metals complexes which are also referred to as coordination complexes" are complexes that contain ions or molecules linked or coordinated to a transition metals. For example, $Fecl_3$ ion and $\{CO(NH_3)_6\}Cl_3$.

The ions or molecules that bind to transition metal ions to form these complexes are called ligands”. The ligands are bound to a metal ion by a coordinate covalent bond (donating electron from a lone pair electron into an empty metal orbital) and are said to be coordinated to the ion. Although coordination complexes are peculiar to the transition metals but some main group elements also form complexes such as aluminum in AIF_3

Alfred Werner, an inorganic chemist developed a model of coordination complexes which explains the following observation

At least three different cobalt (III) complexes can be isolated when $COCl_2$ is dissolved in aqueous NH_3 and then oxidized by air to +53 oxidation state and fourth complex was made by slightly different techniques

$COCl_{3.}\ 6NH_3$	Orange-yellow
$COCl_{3.}\ 5NH_3$	Red
$COCl_{3.}\ 5NH_3$	Purple
$COCl_{3.}\ 4NH_3$	Green

- The reactivity of the NH_3 in these complexes ha been drastically reduced. The ammonia reacts rapidly with hydrochloric acid to form ammonium chloride and these complexes itself

$NH_{3\{aq\}} + HCl_{\{aq\}} \rightleftharpoons$ (2$NH^{+}_{4\{aq\}}$ + $Cl^{-}_{\{aq\}}$ do not react with hydrochloric acid

Even at 100°C

Solution of the Cl^- ion react with Ag^- ion to form a white precipitate of AgCl

$Ag^+ + Cl^- \rightleftharpoons Agcl_{(s)}$

When excess Ag^+ ion is added to the solution $COCl_3.6NH_3$ and $COCl_3.5NH_3$ complexes in solution. However, only two of the Cl^- ion $COCl_3.4NH_3$ can be precipitated with Ag^+ ions

- Measurement of the conductivity of aqueous solution of these complexes suggest that the $COCl_3.6NH_3$ and $COCl_3.5NH_3.H_2O$ complexes dissociate in water to give a total of four ions.

$COCl_3.5NH_3$ dissociates to give three ions and $COCl_3.4NH_3$ dissociated to give only two ions.

Werner explained these observations by suggesting that transition-metal ion such as the Co^{3+} ion has a primary valence and secondary valence. The primary valence is the number of negative ions needed to satisfy the charge on the metal ion, for example, in each of cobalt (III) complexes, three Cl^- ions are needed to satisfy the primary valence of the Co ion. The secondary valence is the number of ions of molecules that the secondary valence of the transition metal in these cobalt(III) complexes is six. The formula of the compounds can be written as follows

$\{Co(NH_3)_6{}^{3+}\}Cl^-)^3$	Orange – Yellow
$\{Co(NH_3)_5)H_2O^{3+}\}Cl^-\}^3$	Red
$\{Co(NH_3)_5)(Cl^{2+})Cl^-)^2\}$	Purple

The cobalt ion has a coordination number of six, that is, a total of six ligands in each complex which satisfies the secondary valence of this ion. Each complex has a total of three chloride ions that satisfy the primary valence. Some of the c1 ions are free to dissociate when the complex dissolves in water. Werner postulated the definite shapes of the transition metal complex which he called the ligand in six coordinate cobalt [111] complexes an octahedral shape.

1.4 LITERATURE REVIEW ON CITRIC ACID METAL COMPLEX

Dimitr et al (11) synthesized and characterized yttrium-iron (III) citric acid complexes with mole ratio Y: Fe =1or 3 :5,potential precursors for $yfeO_3$ and $y_3fe_5O_{12}$ thin films preparation, was synthesized in ethylene glycol medium. The compositions, IR, 1H and 13C NMR spectral characteristics of the isolated solid complexes also studied [10]. Their chemical nature and the peculiarities of the complexation process in the system YIII-FeIII-citric acid-ethylene glycol= 1: 1: 8.5 :35 were studied and compared with those of the earlier studied lanthanide (111 or IV) titanium (IV) citric acid complexes, prepared under the same conditions.

The mixed-metal nature of the obtained complexes has been shown. In contrast to the analogous lanthanide-titanium system, no deprotonation of the alcoholic OH groups of citric ligands takes place in the course of the formation of Y-Fe complexes. [12]

The citric precursor technique is widely used [in aqueous or ethylene glycol solution] to produce a number of multi-component, phase-homogeneous materials, including yttrium-iron garnet ($Y_3Fe_5O_{12}$, YIG), other $Ln_3Fe_5O_{12}$ The authors pay attention to the precursors' composition but point to the necessity of a much better knowledge of their chemical nature. Significant efforts have been made in the last years in this direction by studying the chemistry of the processes involved in $BaTiO_3$ and $Ln_2Ti_2O_7$ production in ethylene glycol [EG] medium by the polymerized complex method [PCM].

However, the reasons for the citric ligands' alcoholic OH group deprotonation, taking place in the course of the complexation process in (Ba or Ln)-Ti-[CA]-EG systems [CA, citric acid] at an uncommonly low PH, have not been fully elucidated. The role of the overall charge of the complexing agents in this process was also studied by substituting Ln111 with CeIV in Ln –TiIV-CA-EG system. It was interesting to compare these results with the ones obtained by substituting the other complexing agent

(Ti4+), as a partner of Ln111, with another metal ion in oxidation state III, like Fe3+are suitable starting materials for YFeO3 and YIG film deposition.[13]

The authors made use of infrared, 1H and 13C spectra and suggested that mixed YIII-FeIII citrates are formed at 100 °C in the Y3+-Fe3+-CA-EG systems but their composition and the way of bonding are different from those of the analogous YIII-TiIV bimetallic citric complexes, prepared under similar conditions. The resorted results contribute to further elucidation of the chemistry of the processes taking place in the application of the polymerized complex method and especially of the mechanism of deprotonation of the citric ligands alcoholic OH group. The Y: Fe mole ratios in the prepared complexes are rather close to the desired ones and, from this point of view, they are suitable starting materials for $YFeO_3$ and YIG film depositions. [14]

CHAPTER TWO

2.0 MATERIALS USED

2.1.1 LIGAND

The ligand used is citric acid monohydrate

- Appearance: white powder with water of crystallization
- Odour: sweet smell
- Colour: white
- Molecular weight: 210g/mol
- Molecular formular: $C_6H_8O_7$

2.1.2 METAL SALTS

The transition metal salt used are ;

Copper acetate ($Cu(CH_3COO)_2$) AND Pb Nitrate ($Pb(NO_3)_2$).

EXPERIMENTAL PROCEDURE

2.1.3 REAGENTS

1. Distilled water
2. Ethanol
3. Acetone
4. Methanol

The above metal salts and regents were obtained from Lab Trade, Ibrahim Taiwo road Ilorin. The chemical compounds used are of analytical grade and were used without further purification.

2.1.4 APPARATUS

Reflux apparatus, Retort stand, Clamp, Water bath, Hot plate, Conical flask, Hose, Test tubes, Filter paper, Funnel, Beaker, melting point Apparatus, Capillary tube and measuring cylinder.

2.1.5 SYNTHESIS OF THE COMPLEXES

GENERAL PROCEDURE

8.57mmol of citric acid monohydrate was reacted with 4.37mmol of metal salt. 5ml of distilled water is then added to the resulting solution. The resulting solution was refluxed and the cool. The complex formed is ten filtered, dried and kept properly

The following are the precautions taken during the synthesis of the complexes

1. All apparatus were thoroughly washed and prior to use
2. An accurate measurement of weight of the ligand and that of the metal salt were carried out
3. The hot plate was properly regulated

4. Conical flasks are washed with the substance to be filled in it.

Synthesis of the complexes were done in the following ways;

1. All apparatus are properly washed
2. All set up were done. The retort stand and clamp were used to arrange the condenser in a vertical position in order to be suitable for the refluxing process. The hot plate was placed directly under the water bath in which the condenser was fitted. The water tubes were fitted into the reflux apparatus (condenser) to continue a counter current cooling
3. Certain amount of the metals salt was weighted using a meter balance and pour into a clean conical flask. A known volume of solvent was added to make up the solution.
4. A certain amount o0f ligand was weighed and placed in a conical flask which was dissolved in an appropriate solvent.
5. The amount salt solution was poured into the ligand solution. Changes such as colour, precipitation, crystal formation were noted and recorded before and after shaking the mixture.
6. The flask was fitted into the opening of the reflex set up and placed in a water bath. The bath was placed on the hot plate
7. The hot was switched on and properly regulated

8. The time taken for the mixture to reflux was also noted and recorded and the final colour of the mixture was noted and the conical flask was removed from the reflux condenser
9. The complexes (crystal or precipitate) obtained was then filtered with the aid of a filter paper.
10. The complexes obtained were allowed to dry at room temperature noting its colour, appearance and then put in a clear labelled container

2.2 SYNTHESIS OF CU{(CA)CH_3(COOH)}$_2$

8.57mmol (1.80g) of citric acid was dissolved in 20ml of ethanol. 4.37mmole (0.80g) of CU{CH_3(COOH)}$_2$. The two solutions were mixed together after which 5ml of distilled water was added to the mixture of the two solutions.

Instant complex of CU{(CA)CH_3(COOH)}$_2$ was obtained after the addition of 5ml of distilled water. The blue precipitate obtained was filtered, dried and kept properly.

2.3 SYNTHESIS OF Zn(CA)(CH_3COOH)$_2$

8.57mmol (1.80g) of citric acid was dissolved in 20ml of ethanol. 4.37mmole (0.95g) of Zn(CH_3COOH)$_2$ was also dissolved in 20ml of distilled water. The two solution were mixed together followed by he addition of 5ml of distilled water. The resulting solution obtained was refluxed for two hours. A white complex of $Zn(CA)(CH_3COOH)_2$ obtained which was filtered, dried and kept properly

CHARACTERIZATION METHODS

The complexes obtained from the experiment were analyzed in the process and characterized using both physical and instrumental method of analysis.

2.4 PHYSICAL CHARACTERIZATION

SOLUBILITY

The solubility of the complex was determined using the following solvent:

Ethanol, Methanol, Distilled water and acetone

A small quantity of the complex was introduced into the test – tube. The solvent for the determination was determined and the observation noted. The complex that was insoluble when cold was heated in a water bath and the solubility was observed

2.5 INSTRUMENTAL CHARACTERIZATION

2.5.1 MELTING POINT DETERMINATION

Melting point of the complexes was obtained using the apparatus:

Capillary tubes

Gallen clamp

Melting point apparatus

Procedure: one end of the capillary tube was sealed and a small quantity of the complex was introduced into the capillary tubes and then placed in the hole of the melting point apparatus. The apparatus was set into operation and the sample was viewed from the lens on the apparatus and the temperature at which the complex starts to melt was recorded

2.6.2 ULTRA-VIOLENT SPECTROSCOPY (UV)

Ultra-violent spectroscopy gives the transition state as well as the visible absorption band of the complexes. This is also carried out at Lautech- Ogbomosho

2.6.3 INFRARED SPECTROSCOPY (IR)

Infrared spectroscopy helps in analyzing and showing various functional groups present in the complex. This plays a major role in the structural elucidation of the complexes. The scanning method was done with the aid of KBr pellet for all the complexes. This was done at Lautech- Ogbomosho.

CHAPTER THREE

3.0 RESULTS AND DISCUSSION

3.1 TABLES OF RESULTS

TABLE 1: RESULTS OF SOLUBILITY TEST.

Solvents	Ethanol	Methanol	Acetone	Distilled water	Acetic Acid
Ligand complex					
Citric Acid	S ‘	S	NS	S	NS
Pb $(CA)(N0_3)_2$	NS	NS	NS	NS	NS
$Cu(CA)(CH_3COO)_2$	S	S	NS	NS	NS

`

TABLE 2: RESULT OF INFRARED SPECTRA

Ligand/ complex	Ʋ(C-H)cm^{-}	Ʋ(O-H)cm^{-}	Ʋ(C=O)cm^{-}	Ʋ(C-O)cm^{-}
Citric acid	1398.02	3384.96	1732.14	______
$Pb(CA)NO_3$	1329.38	3423.72	1642.57	1091.36
$Cu(CA)(CH_3COO)_2$	1306.35	3458.15	1616.32	1082.99

TABLE 3 RESULT OF UV- VISIBLE SPECTRA

Complex ligand	**Wavelength**	**Assignment/ Transition**
Citric acid	**214nm**	**n__** π^*
$\mathbf{Pb(CA)N0_3}$	**209nm**	**n__** π^*
$\mathbf{Cu(CA)(CH_3COO)_2}$	**199nm**	**n__** π^*

3.2 DISCUSSION

In the solubility table, the ligand is soluble IN Ethanol and Methanol but the complexes formed are insoluble in most of the polar solvents which show that coordination has actually taken place.

It can be seen from table 2 that is melting point of citric acid ligand and that of the complexes are different which indicates that coordination occur between the metal salt and ligand.

In table 3, that is, the atomic absorption spectroscopy table, we can see that the percentage of metal absorbed in the coordination process is actually significant. This confirms that coordination had taken place.

In the infrared spectroscopic data of citric acid, there is a shift in stretch band at absorption frequency of 1732.14cm^{-1} which the corresponding complexes does not have. However, it was observed that the carbonyl deprotonates to give stretch band at absorption frequency of 1091.36cm^{-1} and 1082.99cm^{-1} for Pb(CA)(NO_3), Zn(CA)AcO$^-$ AND Pb(CA)AcO$^-$ respectively. According to the result obtained, it was observed that a C-H stretch band frequency of the comp0lexes does not differ from that of the ligand. Therefore, the most probably site of coordination will be the functional group that are close to each other.

The uv-visible spectra as see from table 4 shows n __ π^* transition. For the citric acid complex, blue shift was observed due to decrease in wavelength of complexes as compared to that of the ligand. This shows that there are transfer of electrons between the metal and the ligand which shows the formation of a complex.

The complex exhibit octahedral geometry. Metals coordinate through the oxygen of the carbonyl of the citric. Citric acid was found to be bidentate.

CHAPTER FOUR

4.0 CONCLUSION AND RECOMMENDATION

4.1 CONCLUSION

Citric acid from table complexes with the transition metals. However, the chemistry of these complexes depend on the metal used.

In $Cu(AS)(CH_3COO)_2$, It was notice that there was an instant formation of crystal when metal salt solution was added to the citric acid solution and the rest were refluxed before crystals were formed.

The citric acid ligand is a bidentate ligand and the complexes formed are octahedral in geometry.

4.2 RECOMMENDATION

since the elucidation and characterization of the isolated complexes were limited to the use of standard analytical methods and only gives the idea of functional groups present. I will therefore recommend that

1) NMR should be included so as to prove beyond reasonable doubt the structure of the complex
2) The federal government should allocate more fund to tertiary institution to acquire more instrument for analysis in order to encourage young researcher for better competence in the world of chemistry standard.

Reference

1.

Penniston W., Nakada S.Y, holmes R.P, Assimos D.G (2008). "Quantitative assessment of Citric Acid in Lemon, Products" (PDF). Journal of Endourology 22(3): 567.

2. http ://www. Islamicspain.tv/Arts and sciences/the culture of –Al-Andalus/Chemistry. Htn
3. http://Muslim Medonat work. Com/mmn/?p=1553
4. http\://journalsoucr.org/a/issues/2008/01/00/Sc5012/index.html.
5. http://web1:caryacademy.org/cheistry/rustin/tudentprojects/compoundwebsites/2001/citric20Acid/historyhtme.
6. http://books.google.com/books?id=ouxom8bdGIVC&pg=PA944=how+citric+acid+was+discovered .
7. Citric acid production by a novel Asergillus niger isolate:II. Optimization of process parameters through statistical experiential designs. Bio resources technology 98(18) 347-3477
8. Garden J., Roberts, K Taylor a., and Robinson D(2083) "Evaluation of the provision of single use citric acid sachets to injecting drug user" (pdt) Scottish Centre for infection and environmental health.

9. Robinson J. (ed). “The oxford companion to wine’ Third Edition pg 171 oxford University press 2006 ISBN 0198609906.
10. Sankaranarayanan. V. K and Gajibhiye N. S., Thermochim. Acta 135 (1989) 337-341.
11. Sankaranarayanan. V. K and Gajibhiye N. S., J. Am. Ceram. Soc. 73 (1990) 1301 -1307.
12. Prasad. S. and Gajbhiye N. S., J Alloys Compd. 265(1998) 87-92.
13. Gajibhiye N. S., . Prasad S, and Balaji G, IEEE Trans. Magn. 35 (1999) 2155-2161.
14. Kakihana m., Milanova M, Arima M., Yoshima M., and Yoshimura M, J Am. Ceram. Soc 76 (1996) 1673-166.