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Green Extraction, Synthesis, Characterization And Anti-Bacterial Activity Of Zinc (Ii) Complex Of Solar Aided Aqueous Extract Of *menthapiperita*(PEPPERMINT)

Japhet Joel Gongden[1], Hannah Amadike[1*], Abu Emmanuel Benjamin[1], Williams Nashuka Kaigama[2]
[1] *Department of Chemistry, Faculty of Natural Sciences, University of Jos, Nigeria.*
[2]*Department of Chemistry, Faculty of Physical Sciences, Ahmadu Bello University, Zaria Nigeria*

ABSTRACT
Complex of Zinc (II) of aqueous solar aided extract of menthe piperita (Peppermint) was synthetized and characterized using UV-Vis, FT – IR and XRF. The antibacterial potency of the complex was assessed. The physicochemical results revealed pH of 10.65, white color, melting point of 220^{0}C , percentage yield of 66.66%, conductivity of 9.49 Zinc (II) complex and soluble in water. The phytochemical screening for the extract and Zn (II) complex indicated the present of alkaloids in both the extract and complex with variating present of the other constituents such as saponin, and flavonoids. The Zinc (II) complex also showed bathochromic shift at 492nm indicating increase in conjugation as a result of ligand to metal charge transfer (LMCT) from $\pi - \pi^r$ orbital. The FT – IR spectra showed that the plant extract and Zn^{2+} complex contain functional group of Amine, Alcohol, carboxylic acid, alkane and alkene. XRF spectra revealed the Zinc content in Zn^{2+} complex as 3.0078% and 0.0639% as the Zn content in the plant extract. The Antibacterial assay revealed as tested on E. coli, P. Aeruginosa, staphyloccusaureus and Bacillus substillus showed that the complex has more potency to inhibit bacteria than the plant extract at various concentration mm/mg (200, 100, 50, 25, 12.5) using Gentamycin as the control at 10mm/mg. The Zn^{2+} complex shows better anti-microbial effect than the plant extract.
Keywords: *Green synthesis, Zinc complex, menthe piperita, antimicrobial.*

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I. INTRODUCTION

Metal complexes have received particular attention for their positive impact on improving many economy sectors. Their properties such properties like high electron mobility, good transparency, high room temperature luminescence, etc. make them find application in consumer products, energy, transportation, cosmetics, pharmaceuticals, antimicrobials agents and agriculture [1]. For example in Medicine it has found great application in drug delivery l2], tissue/tumor imaging [3], antibacterial material [4], anti-diabetic activities [5] etc. It is one of today's most promising and popular fields of scientific research.

The drug resistance of human pathogens is a major theme reported in both developed and developing countries. The daily global consumption of over one ton of antibiotics has led to a large increase in resistance of bacterial populations, thus causing a serious public health problem. The search for new antimicrobial substances has gained importance in pharmaceutical companies and scientific research. Recently, many researchers have been working on the antibacterial activities of metal oxides with growing concern to produce eco-friendly metal complexes, which does not use toxic materials.

Green plants have shown competency to soak up, hyper accumulate and reduce inorganic metallic ions from their surroundings. It is now acknowledged that numerous organic entities existent in plant tissues are capable of performing biological factories to considerably lessen environmental contamination. Moreover, amalgamations of molecules found in plant extracts can behave as both reducing and stabilizing (capping) agents, all within synthesis.

Metal complexes attained from plant extracts are prepared from living plant extracts. Plant parts like leaf, root, latex, seed and stem are widely being used for metal- and metal oxide complexes shows important bio-reductants found in plant extracts. Plant extracts are bioactive polyphenols, proteins, phenolic acids, alkaloids, sugars, terpenoids, etc., which are primarily composed to play a major role in relegating the metallic ions and then alleviating them.

Many plant and plant extracts have been used in the green synthesis of metal complexes. plants such as *ZiziphusMauritiana* L [6],Tribulusterrestris[7],*Thevetiaperuviana* [8], *Meliadubia* [9], *Passifloracaerulea* [10] have been used and the results show that it could be used as a powerful capping agent and as good reducing agent for the synthesis of metal complex.

Menthapiperita is a sterile hybrid of the species M. aquatica L. and M. spicata. This plant is widely used in folk remedies and traditional medicine for treatment of digestive disorders and nervous system actions because of its antitumor and antimicrobial properties, chemopreventive potential, its renal actions, antiallergenic effects, and also for lessening cramping, digestive complaints, anorexia, nausea and diarrhea.[11]. Therefore this research is aimed at extraction, synthesizing, characterizing Zinc (II) complex from solar aided aqueous extract of *Menthapiperita* (Peppermint) and their anti-bacteria efficacy for drug formulation and tackling of drug resistance by bacteria.

II. MATERIALS AND METHOD

2.1 Materials: Distilled water, Hydrogen peroxide, Zinc Nitrate ,Iron (iii) Chloride, Distilled water, Hydrogen peroxide, Zinc Nitrate, Iron (iii) Chloride, Constructed solar aided vessel, Erlenmeyer flask, Oven, weighing balance, Conical flask, Mortar and pistol, Beaker, measuring cylinder, stirring rod, heating mantle

2.2 Sample Collection and Preparation

Plant leaves of the *Menthapiperita* was washed with distilled water, chopped into smaller fraction to open up the surface area of the leaves. The chopped leaves were placed in the constructed solar vessel alongside distilled water; the vessel was place under the sun so as to allow interaction between sunlight and the mixture in the vessel (plant and water).

After each day, the water extract was collected and replaced with another for effective extraction and this was carried out for five days. The water extract for each day were mixed together, concentrated and grinded to powder.

2.3 Synthesis of Zinc Complex

10g of the powdered extract was dissolved in 50ml of distilled water and filtered. 5g of the metal salt of Zinc Nitrate was also dissolved in 50ml of distilled water. The dissolved Zinc Nitrate was added to the filtrate respectively with observed color change. 2ml of Hydrogen Peroxide was added to the mixture respectively for reduction Zinc ions and then heated at 50^oC for five (5) minutes with observed color change and cooled immediately. The mixture was allowed till the next day for nucleation and evaporation. The solid complexes formed for Zinc was properly kept for characterization and other chemical analysis.

2.4 Characterization of Zinc Complex

The reduction of Zinc nitrate to Zinc ion by the pepper mint extract was determined by it color, melting point, percentage yield, UV-Vis spectroscopy, the determination of the functionality or the functional groups present in the plant extract and that of the complexes formed using Fourier Transform Infrared spectroscopy (FT-IR), XRF (X-ray fluorescence) and XRD (x-ray diffraction) for the complexes were determined to give its composition.

2.5 Anti-bacterial assay

The antibacterial activity of the plant extract, Zinc complex was developed at different concentration and evaluated on four different types of bacteria (Gram-positive and Gram-negative) with the assessment performed using the Well Diffusion method.

III. RESUSLT AND DISCUSSION

3.1 Characterization of *Menthapiperita* leaf extract and green synthetized complex

3.1.1<u>Phytochemical Analysis for the Plant Extract</u>

Table 3.1 indicates that *Menthapiperita* leaf is rich with Tannins, Alkaloids and Flavonoids.

Table 1: Phytochemical Analysis for the Plant Extract and Complexes.

Constituents	Plant extract
Alkaloids	+
Saponins	+
Tannins	+++
Flavonoids	+++
Carbohydrate	+
Steroids	-
Terpenes	-
Anthraquinone	-

Cardiac glycosides	-

Key: + means present, ++ means moderately present, +++ means highly present and – means absent

3.1.2 <u>Physicochemical Parameters for the green synthetized Complex.</u>

Table 2

Parameter	**Zn-Peppermint Complex**
pH	10.65
Conductivity	9.49
Color	White
Melting point	220^{o}C
Solubility in Water	Soluble
Solubility in Ethanol	Slightly soluble
Solubility in Methanol	Slightly soluble
Solubility in Propanol	Slightly soluble

3.2 FT - IR ANALYSIS

In order to determine the functional groups of*Menthapiperita* leaf extract and to identify their role in the synthesis of Zn ion complex, FTIR analysis was performed (Fig. a and b). Leaf extract FTIR showed a band at 327240 and 2922 cm$-^{1}$, which have been due to O−H stretching of alcohol or phenol and C-H stretching of alkane. 1401.5 cm^{-1} stretching show the presence C-C of Aromatic ring while the adsorption peak at 1252.4cm^{-1} is attributed to C-N stretch of Aromatic Amine.FTIR spectra of green synthesis Zn complex have shown absorption band at 3451.5/3213.0cm$^{−1}$, which is a characteristic signal of the alcohol and phenol, while, the bands at 1457.4, 1408.9 and 1084.7 cm$^{−1}$ are attributed to the alkane, aromatic rings and Aliphatic amines functional groups present in the organic compounds respectively. From the FTIR result the soluble elements present in *Menthapiperita* leaf extract could have acted as capping agent preventing the aggregate of the metal particles in solution and playing a relevant role in their extra cellular synthesis and shaping.{12]. These compounds in the plants may be responsible for the reduction of the metal salt.

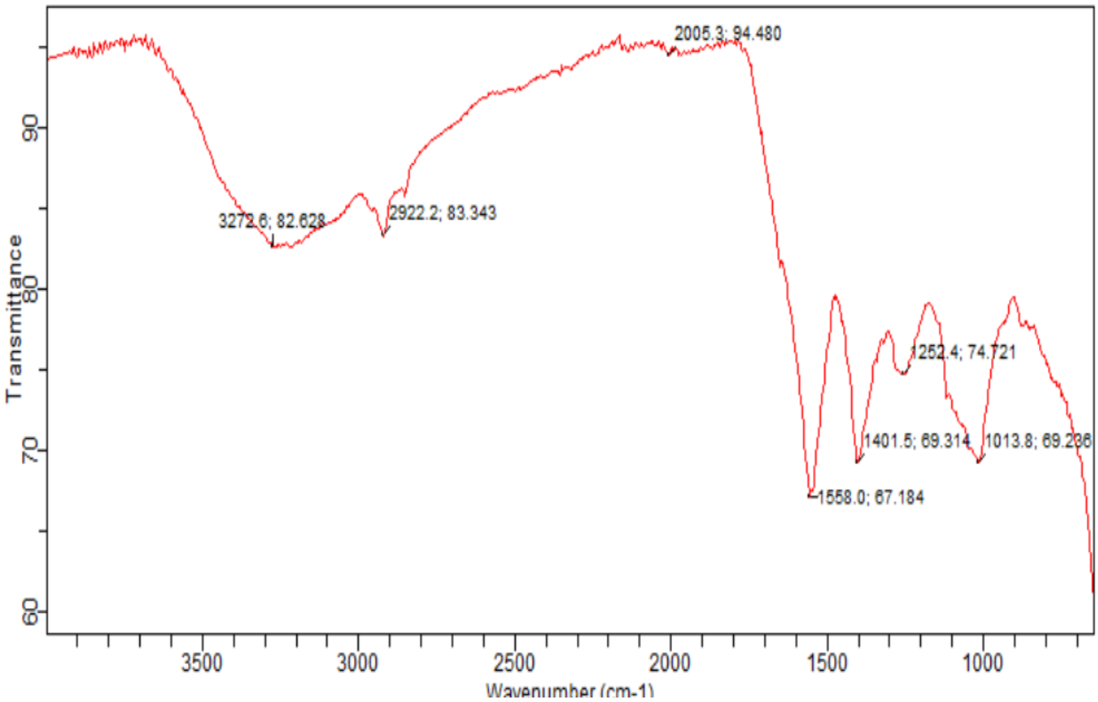

Fig 1.FT IR spectra of pepper mint

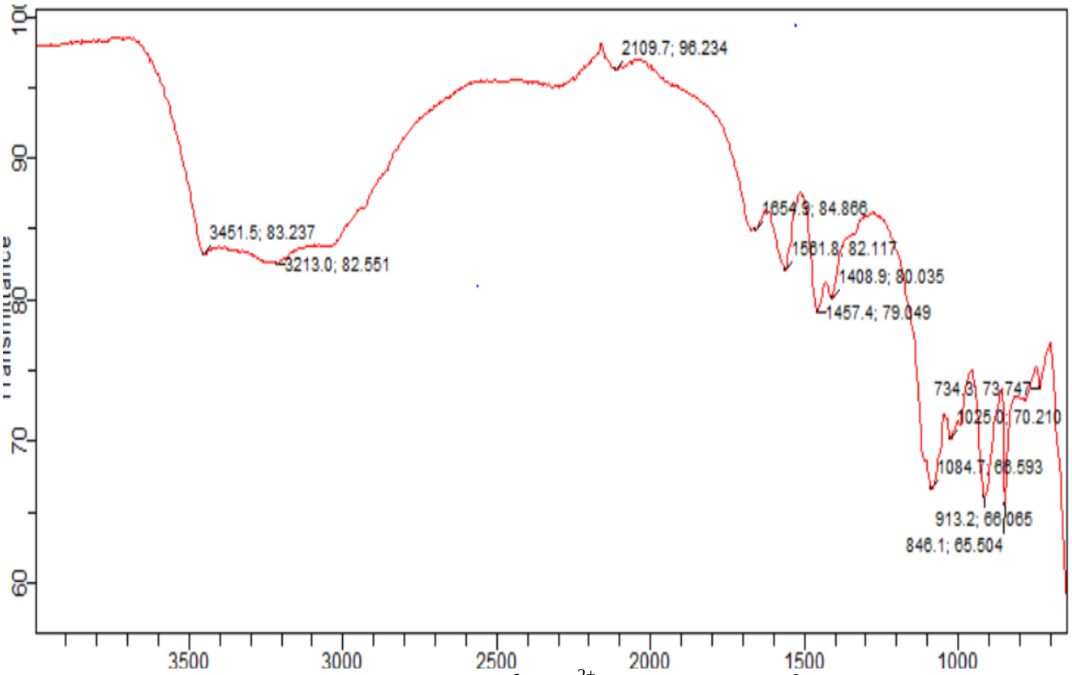

Fig 2. FT-IR Spectra for Zn^{2+}-Peppermint complex

3.3 UV–vis spectrum: The properties of biosynthesized Zn-pepper mint complexes were investigated using UV–Vis absorption methods. A sharp absorption band around the wavelength of 360 nm is clear in UV–Vis absorption spectrum which falls within the absorption peak maximum for Zn complexes ranges between 300 and 380 nm. [13]. This adsorption band connotes a shift to higher wavelength in the maximum wavelength of the complex indicating complexation of the ligand toward the metal ion confirming coordination of the ligand to the metallic ion. This wavelength for the complexes could be assigned to ligand to metal charge transfer (LMCT).

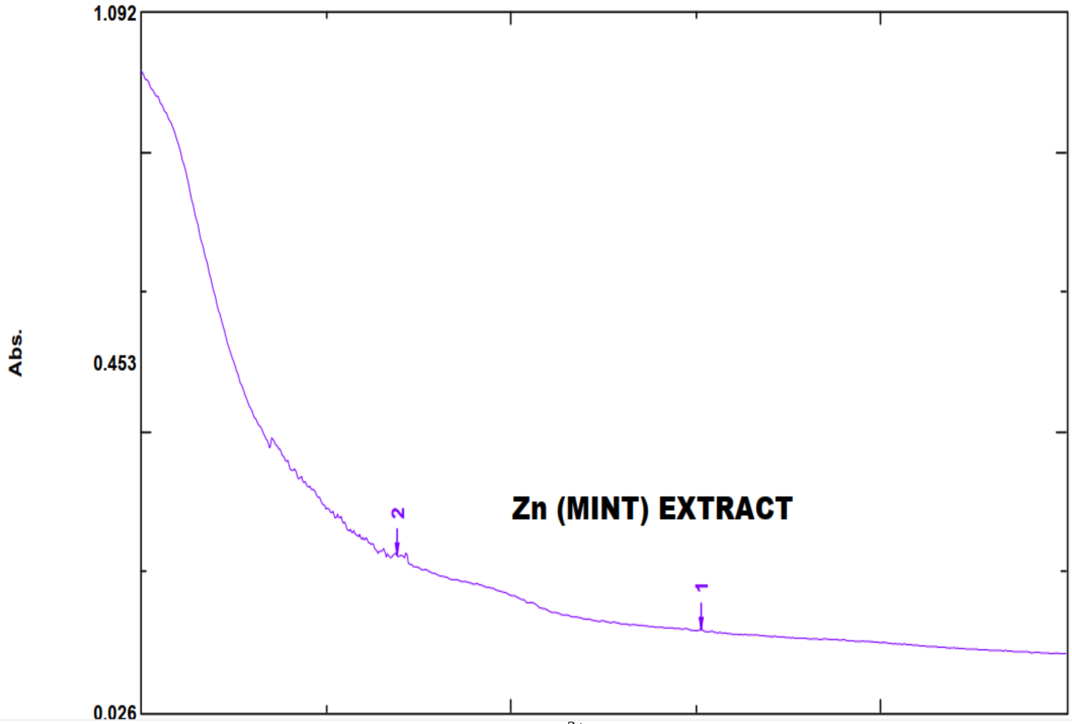

Fig 3.UV-VIS Spectra for Zn^{2+}-Peppermint complex.

3.4 XRF (X-RAY FLUORESCENCE)

Table 3 Shows great disparity in the content of the different common element present in the plant extract and the complex formed. The Zn, Silicon and Phosphorus content in the extract experience a great increase in the Zinc-peppermint complex content. Therefore, in the Zinc-peppermint complex, the Zinc content in the Zinc-peppermint complex is greater than that of the extract which is greater than of Iron-peppermint extract. The changes in the content of the various elements are as a result of the excitation of the element present in the plant extract by bombardment with high x-ray energies. Indicating the incorporation of the metals into the extract to form the desires complexes with support from the other spectroscopic techniques used.

Table 3 : Elemental Content Present in the Plant Extract and Complexes

Element present %	Extract content	Zinc-peppermint complex content
Mg	0.8509	-
Al	7.6463	6.4656
Si	7.8935	7.8749
P	1.8585	5.9743
S	0.4108	-
K	24.3634	0.4670
Ca	6.5547	0.1534
Fe	0.1551	0.1233
Zn	0.0639	3.0078
Sr	0.0100	-
Nb	0.0158	0.0256
Mo	0.0040	0.0333
Cd	0.0141	0.0306
Sb	0.0119	0.0144

3.5 ANTI BACTERIAL ASSAY.

The antibacterial assay was carried out at five different concentrations (12.5-200mm/mg) and tested on two gram-positive bacteria (*Staphyloccusaureus* and *Bacillus subtillis*) and two gram-negative bacteria (*Pseudomonas aeruginosa* and *E. coli*) using the plant extract and the complex formed

Both Gram positive as well as Gram negative bacteria shows excellent activity towards biosynthesized Zinc complex compare to the pepper mint plant extract as shown in table 3.5a & b .

The sensitivity test helps find out the most effective sample on the different bacteria used that is the test determines the susceptibility of bacteria to antibiotic or the ability of the samples to kill the bacteria thereby reducing bacteria resistance to sample or antibiotic.From the sensitivity table above, the Zn-Peppermint complex shows high susceptibility to the bacteria in comparison to the plant extract.

Overall observations indicate that green synthesized Zn complex can beuseful and effective agent for drug formulation and control of bacterial pathogens, which will be more specific and cost-effective.

Table 4 Minimum Inhibitory Concentrations (MIC)

Samples	**Microorganism (Bacteria)**	**MBC Concentration Mg/ml**					
		200	**100**	**50**	**25**	**12.5**	**MBC**
A. Peppermint Extract	*P. Aeruginosa*	-	-	-	+	+	50
	E. Coli	-	-	+	+	+	100
	Staphyloccusaureus	-	-	-	+	+	50
	Bacillus subtillus	-	-	+	+	+	100
B. Zinc Complex	*P. Aeruginosa*	-	-	-	+	+	50
	E. Coli	-	-	-	+	+	50
	Staphyloccusaureus	-	-	-	-	+	25
	Bacillus subtillus	-	-	-	+	+	50

Key: MIC= Minimum Inhibitory, - = No Turbidity, + = There is Turbidity

Table 5 Bactericidal Concentration of Samples

Samples	Microorganism (Bacteria)	MBC Concentration Mg/ml					
		200	**100**	**50**	**25**	**12.5**	**MBC**
A. Peppermint Extract	*P. Aeruginosa*	+	+	+	+	+	-
	E. Coli	+	+	+	+	+	-
	Staphyloccusaureus	-	+	+	+	+	200
	Bacillus subtillus	+	+	+	+	+	-
B. Zinc Complex	*P. Aeruginosa*	-	+	+	+	+	200
	E. Coli	-	+	+	+	+	200
	Staphyloccusaureus	-	+	+	+	+	100
	Bacillus subtillus	-	-	+	+	+	200

Key: - = No Growth, + = there is growth on the plate with microorganism, MBC = this is the final concentration that kills the microorganisms.

IV. CONCLUSION

The investigation demonstrated the synthesis of zinc complex using a simple, eco-friendly and green chemistry approach. *Menthe piperita* (Peppermint) leaf aqueous extracts have been used as a reducing and capping agent for the synthesis of zinc complex. Several techniques, namely UV–Vis spectroscopy, FTIR analysis and XRF have been utilized in this investigation to analyze both the property and quality of the biosynthesized Complex formed. The results of antimicrobial activities revealed that maximum zones of inhibition was observed Gram positive bacteria and followed by the Gram negative bacteria at concentration of 200 *m*g/mL of Zn complex. The above results show that the biosynthesized Zn complex can offer a significant contribution to many biological processes and in pharmaceutical industries for drug formulations in tackling the problem of drug resistance caused by bacteria.

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