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(RESEARCH ARTICLE)

Synthesis, characterization and bioactive evaluation of FE- CO nanoparticles from the leaf extract of *Mentha piperita* (Pepper Mint)

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Abstract

Investigations on some bioactive Fe and Co Nano Particles as bimetallic Fe-Co nanoparticles were synthesized using *Mentha piperita* leaf extract as reducing agent. Hydrated Co and Ni reagents were utilized as metals precursors. The study was aimed to synthesized Nano Particles and characterized them using UV-Visible spectrum which showed Fe-Co Nps highest absorbance at a peak of 400nm. FTIR of leaf extract revealed that 3432cm^{-1} (O-H), 2936 cm^{-1}(C-H) stretching, 1638 cm^{-1}(N-H) 1340 cm^{-1} (N-O) and 659.38 cm^{-1} (C-C) functional groups, while Fe-Co Nps showed the same with addition of 1694 cm^{-1} (C=O) and 666.04 cm-1 (C-C) triple bond. XRD analysis showed peaks to the plane (111), (110), (211) and (220) representing face centered cubic (FCC) structure whose size of 52.64nm. SEM analysis showed irregular cubic and hexagonal shape of different sizes. Antibacterial activity of Fe-Co Nps showed increase in bacterial growth in *Eschemicha-coli* while *psendomonas aureginos* (Gram-negative) and *Klebsella pneumonia* (Gram-positive), showed inhibition with increase in concentration of the synthesis drug. Antifungal activity test on Fe- Co NPs revealed that *Aspagillus niger* shows an increase growth inhibition with increase in concentrations of synthesis drugs the process of activity was the same in case of Candida fungi

Keywords: Nano particles; Extract; Wavelength; Spectra; Concentrations

1. Introduction

Integration of green chemistry principles to nanotechnology plays a vital role in nanoscience research. Biological methods were used to synthesize metal and metal oxide nanoparticles of specific shapes and size since they enhance the properties of nanoparticles in greener route. Plant-mediated methods avoid the use of toxic chemicals in the synthetic protocols which has adverse effects on the environment [1]. Owing to the rich biodiversity of plants and their potential secondary constituents plant and their parts gain attention nowadays as medium for nanoparticles synthesis. Green synthesis of nanoparticles nowadays has gain significant attention to protect environment from hazardous waste. [2]. Green synthesis was design for chemical products and processes that eliminate applications hazardous substances [3]. It is also an emerging area in the field of nanotechnology that provides alternative economic and environmental benefits to ecosystem.

Modern technology on experimental research for synthesis of metal nanoparticles require morphologies and sizes have become a major focus of researchers [4]. Synthesis of metal nanoparticles using organism has emerged as a non-toxic and suitable for ecosystem in the synthetic processes. Green Chemistry upholds diverse areas of wisdom in physical properties, reactions of materials used as investigated by the Scientist. Nevertheless, the notion 'green' is not a quantitatively established parameter, but it tends to be distorted the intuitive response to green. It is not just living organisms that are green in color but rather encompasses quantity, human safety, long term effects, and acute toxicity. Green is a term associated with a sustainable deviations product or non-toxic process whose enrollment in society has no acute toxicity and a general favorable life cycle analysis. Several chemicals are toxic in nature even at negligible does which are annulling the invalidation of green synthesis [5].

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2. Materials and Methods

2.1. Sample Collection

The matured *Mentha piperita leaves* were collected from Filiya town in Shongom Local Government Area, Gombe State, Nigeria and transported in a brown envelop to Gombe state University. It was identified and authenticated by the University herbarium section of Botany Department as adopted by [6].

2.1.1. Sample preparation

The matured *Mentha piperita* samples collected were washed four times with tap water and then rinsed with distilled water to remove adhered dirt and then air dried, ground using a wooden mortar and pestle and sieved into powdered for extract preparation as adopted by [6].

2.1.2. Extract Preparation

Methods adopted by [7] were slightly modified whereby, 20 g of the powdered (sample) was weighed and dispersed into 400 ml of distilled water in 500 ml glass beaker and boiled at 80 ^{o}C for 60 minutes and then allowed to cool and filtered using whatmann filter paper and the filtrate was use immediately for the synthesis of the nanoparticles.

2.1.3. Preparation of $CoCl_2.6H_2O$ Solution

Exactly 0.066 M solution of $CoCl_2.6H_2O$ was prepared by dissolving 8.46 g of $CoCl_2.6H_2O$ salt into 1000ml volumetric flask, and distilled water was added up to the mark as described *by* [7].

2.1.4. Preparation of $FeCl_3$ Solution

$FeCl_3$ solution was prepared by dissolving 1.6231g of $FeCl_3$ salt into 1000 ml volumetric flask, and distilled water was added up to the mark as described by [7].

2.2. Synthesis of Fe-Co Bimetallic Nanoparticles (Fe-CoNPs)

Adopting modified method of [7] for the synthesis of iron and manganese nanoparticles. The prepared $FeCl_3$ and $CoCl_2.6H_2O$ solutions were added drop wise into the plant extract in a ratio of 1:5 with constant stirring at 60 ^{o}C for 60 minutes using magnetic stirrer. Within the first 15 minutes the color change was observed which indicated the formation of nanoparticles and then aged and allowed to settle for 24 hours (one day) after which it was decanted. The collected nanoparticles were dried at 100 ^{o}C for 2 hours It was then ground into powdered for further analysis.

2.3. Characterization Methods

2.3.1. UV-Visible Spectrophotometry Analysis

UV-Visible spectroscopy analysis was done to confirm the formation of nanoparticles and observe the extract's plasmon vibration and excitation. The wavelength was varied at regular wavelength intervals of 200 nm, 300 nm, 400 nm, 500 nm, 600 nm, 700 nm and 800 nm respectively as adopted by [6].

2.3.2. Scanning Electron Microscopy (SEM) Analysis

The SEM analysis was done in order to determine the morphology, size and the composition of the element present in the synthesized Fe-Co bimetallic nanoparticles. It was done using powdered sample in question as adopted by Elisha, *et al.,* [8]

2.3.3. Fourier Transform Infrared (FTIR) Analysis

FTIR analysis was done on the leaf extract, Perkin Elmer Spectra Version 1003 09 machine. The synthesized Fe-Co, bimetallic nanoparticles were investigated which determine the functional group present in the sample as adopted by [8]

2.3.4. X-Ray Diffraction (XRD) Analysis

XRD analysis was done using *X-Pert Plus machine* which determined the average crystalline size of the nanoparticle samples. The Debye scherer equation was used to calculate the average crystalline size. The Debye scherer equation is as follows

$$D = \frac{K\lambda}{\beta COS\theta}$$

Where
D= Particles size = 0.94
K= Constant volume
λ= X-ray wavelength(0.154nm)
P=Line broadening at half the maximum intensity
θ= Braggs angle (in degree) as reported by Elisha, *et al*[8]

2.4. Antimicrobial Activity Test

2.4.1. Media preparation

The media preparation depends on the manufacturer's specification. 28 g of nutrient agar was dissolved into 1000 ml deionized water and autoclaved at 121 ^{o}C for 15 minutes to sterile as adopted by [8]

2.4.2. Sensitivity Test

This test was conducted by incubating the bacteria and fungi overnight at 37 ^{o}C and the zone of bacteria and fungi growth inhibition was measured in millimeter (mm) as adopted by [9].

2.4.3. Antimicrobial Activity Studies

The antimicrobial activity studies were carried out by using the agar well method to test the antimicrobial activity of the green synthesized Fe-Co, bimetallic nanoparticles on two gram negative bacteria, Escherichia-*coli and pseudomonas aureginosa,* two gram positive bacteria, *Staphylococcus aureus and klebsella pneumonia,* and two fungi, *Aspagillus Niger and Candida*. This was conducted by creating 6 mm hole in the prepared agar (media) inside the petridish. The organism was inoculated all over the surface of the petridish and the synthesized drugs (nanoparticles) were also inoculated in to each hole with a control drug at the center. It was then incubated overnight at 37 ^{o}C after which the zones of bacterial and fungal growth inhibition were measured in millimeters (mm) as adopted by [9].

3. Results and Discussion

3.1. UV Absorption Wave Length of Nanoparticles

Figure 4.6 showed Fe-CoNPs UV absorption wavelength with the highest absorbance was observed around 400 nm indicating the formation of Nps due to the excitation of the surface plasmon vibration in the Fe-CoNps. However, similar work done by [10] talked about the possibility of finding the plasmon band of AgNPs in range from 400-420 nm, also, the work is similar to findings by [11] and [12]. Thus, the result obtain was contrary the 300 nm and 650 nm as investigated by [6].

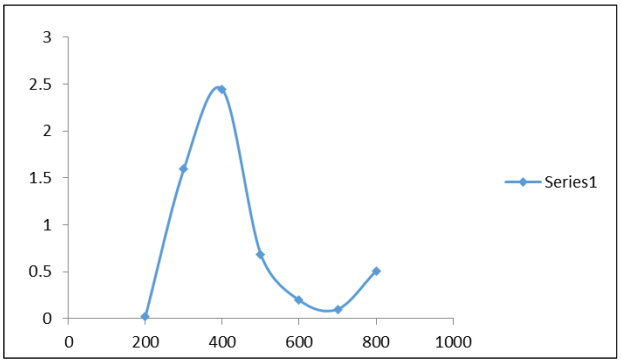

Figure 1 U.V. spectrum for Fe-Co NP

3.1.1. FT-IR Spectra for Mentha piperita Leaf Extract

Figure 4.8 showed the *Mentha piperita* extract's FTIR spectrum displayed wave length values with several peaks, at 3432.19 cm^{-1} represents O-H free hydroxyl and H-bond due to alcohol and phenols. 2936.13cm^{-1} indicate C-H stretches due to alkanes, 1638.16 cm^{-1} indicate N-H bond due to primary amine, 1340.29 cm^{-1} N-O symmetry due to nitro

compound. 659.38cm^{-1}indicate –C-C_trpple bond: C-H bend from alkynes. 537.18 cm^{-1} indicate C-Br stretch due alkyl halide. This is in agreement with the literature reported by [13]. Which show that, The FTIR bands obtained are characteristics of flavonoids, triterpenes, furanoid, sugar, coumarins, tannins, phenols and acid present in the aqueous extract of *propolis.* The result is synonymous to the findings of [6].

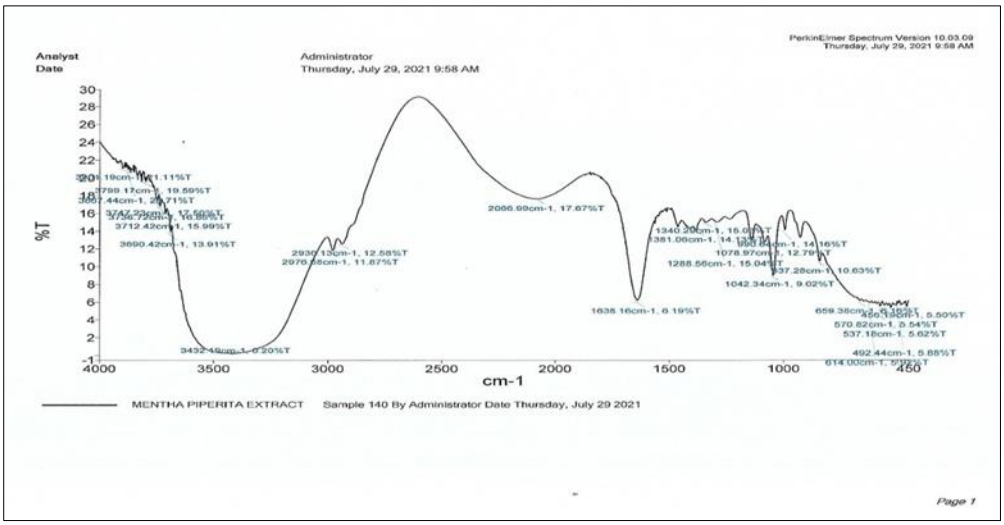

Figure 2 FTIR of Spectra for Mentha piperita Leaf Extract

3.1.2. FT-IR Spectra for Fe-CoNPs

The FTIR spectrum displayed in figure 3 below shows several peaks displayed by Fe-CoNPs. The peaks at 3616.93cm^{-1} and 3624·08 cm^{1} represent O-H stretches free hydroxyl due to alcohols and phenols, 1694.95 cm^{-1} represents C=O stretches free hydroxyl due to α and β unsaturated aldehydes and ketones, 1680.00 cm^{-1} and 1644.58 cm^{-1} represent –C=C- stretch, due to alkenes, 1554.68 cm^{1} representing N-H bend due to presence of 1^{o} amine, 1535.16 representing, N-O asymmetric stretch due to presence of nitro compound, 109-0.00 cm^{1} representing C-N stretch due to aliphatic amines, 666.04 cm^{1} indicating –C-C triple bond: C-H bend due to presence of due to alkynes and 674.68 cm^{1} indicating C-Br stretch due to presence of alkyl halides. Functional presence agreed with the literature reported by[4] which shows that the FTIR bands obtained indicate the present of flavonoids, polyphenols, triterpenes and amide present in the *P.niruri*leaves extract. but the result obtain C- Br was contrary the functional group of C- OH as investigated by [6].

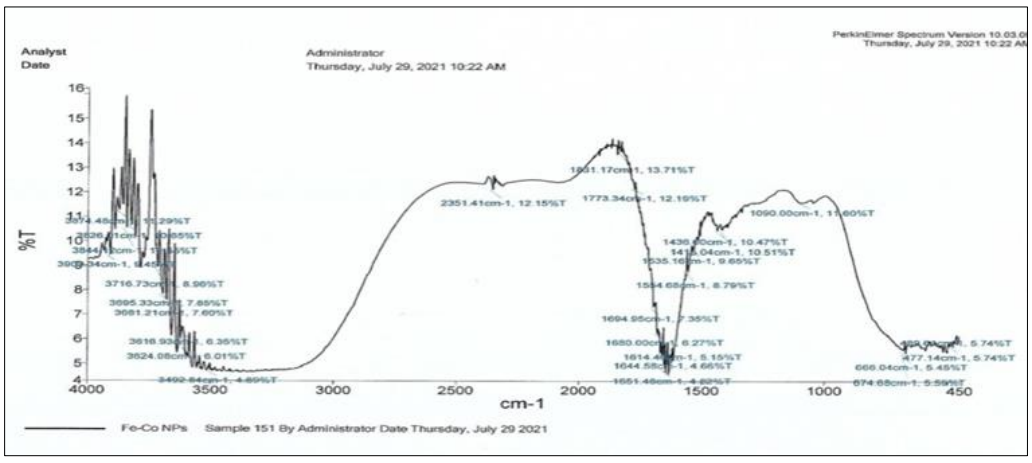

Figure 3 The FT-IR Spectra for Fe-Co NPs

3.2. SEM Analysis

The Morphology of the (Fe-Co NPs) bimetallic nanoparticles of Figure 4.16 below indicated irregular cubic and hexagonal shape of various sizes that are agglomerated. Further observation with higher magnification revealed that these images possess rough surfaces. At higher magnification, the images are seen as large particles which can be attributed to aggregation or clustering of small particles. The result obtain was synonymous to the work done by [14].

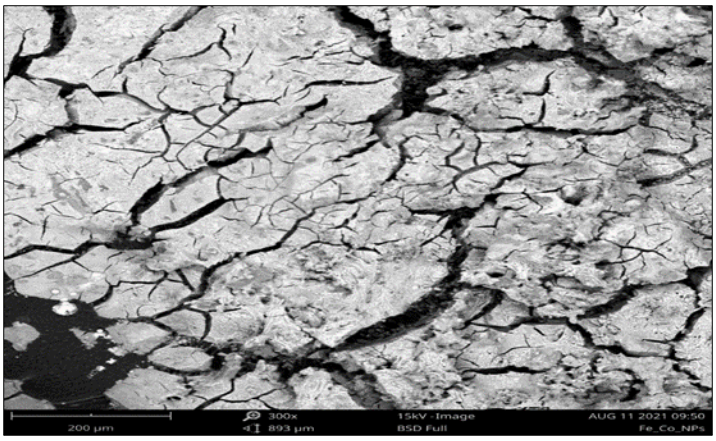

Figure 4 SEM result for Fe-Co NPs

3.3. XRD Analysis for Fe-CoNPs

From the result obtained for the XRD analysis of the Iron-Cobalt bimetallic nanoparticles in fig 5, four prominent peaks were observed at 2θ = 15.86˚, 21.45º, 28.19º and 43.81º. With respect to the plane of (111), (110), (211) and (220), it shows Face Centered Cubic [FCC] structure and the average crystalline size of 52.64 nm. According to the scherer equation calculated. The result obtains corresponded to the literature reported by [15], which shows FCC (face centered cubic) structure for the green synthesized silver nanoparticles using *Tapidium draba* weed extract.

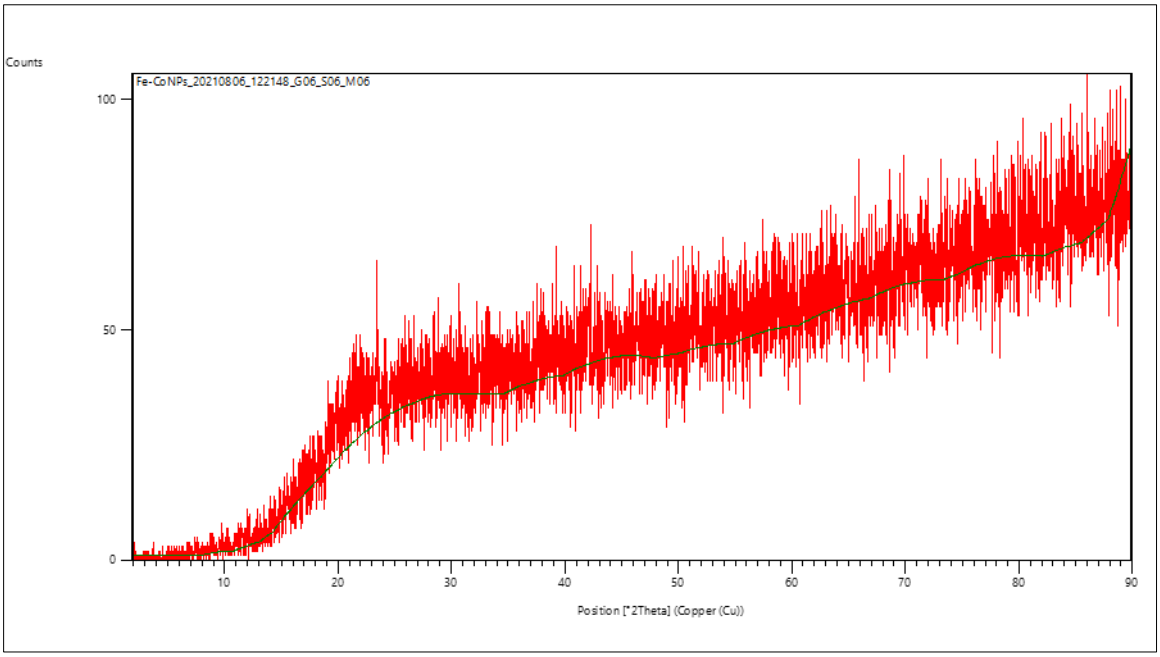

Figure 5 XRD Result for Fe-CoNPS

3.4. Antibacterial Activity Test for Iron-cobalt Nanoparticles

Table 4.1 showed the antibacterial activity test for iron (Fe-Co-NPS) whereby the *Escherichia-coli* show an increase in the bacteria's growth inhibition with increase in concentration of the nanoparticles.

Pseudomonas aureginosa showed an increase inhibition in bacterial growth inhibition with increase in concentration of the synthesized drug (NPS).

Staphylococcus aureus shows an increase in bacterial growth inhibition with increase in concentration of the synthesized drug. *Klebsella pneumonia* also shows an increase in bacterial growth inhibition with increase in concentration of the nanoparticles which are related to the work [6].

Table 1 Antibacterial Activity Test for Iron-Cobalt Nanoparticles (Fe-CoNPS)

Zone of Inhibition in Millimeters (mm)						
Test organism	**100µg/ml**	**200µg/ml**	**300µg/ml**	**400µg/ml**	**500µg/ml**	**Control(300µg/ml)**
E. Coli	7	9	12	14	17	23
P. Aureg	8	9	12	15	18	21
S.Aureu	9	12	15	17	19	25
K.Pneu	12	19	20	22	24	27

Control = *Augomentinem E. Coli* -= *Escherichia Coli*, P. Aureg = *Pseudomonas Aureginosa*, *S.Aureu = Staphyloccocus Aureus, K.Pneu = Klebsella Pneumonia*, µg/ml = Microgram per miNote, each result was obtained by taking the average mean in (mm) where n=2, 6 mm in indicate no activity it only resists the drug, 9mm is weak,16 mm= moderate and 16 above are significant values of activity.

3.5. Antifungal Activity Test for Fe-CoNPS Nanoparticles

By using iron-cobalt nanoparticles [Fe-CoNPS), *Aspagillus niger* shows an increase in fungal growth inhibition with increase in concentration of the synthesized drug (nanoparticles). *Candida*, the same applies to *candida,* it also showed an increase in fungal growth inhibition with increase in concentration of the nanoparticles which is closed to that of the synthesized drugs.

Table 2 revealed Antifungal Activity Test of fungi *candida* and *Aspagillus niger*, where each result was obtained by taking the average mean where n=2. 6mm indicate no activity, it only resists the drug. The test indicated that 9mm is weak, 9-16mm is moderate and 16 above is significant. The result obtained was synonymous to the findings of [6].

Table 2 Antifungal Activity Test of fungi *candida* and *Aspagillus Niger*

Zone of Inhibition (mm)						
Test organism	**100µg/ml**	**200µg/ml**	**300µg/ml**	**400µg/ml**	**500 µg/ml**	**Control(500µg/ml)**
Fe-CoNP^S *A.Niger*	8	10	13	16	19	23
Fe-CoNP^S *Candida*	9	11	13	15	18	24

Control = Fulcin, A.Niger = Aspagillus Niger, Candida = Fungus Candida, µg/ml = Microgram per mil

4. Conclusion

Bimetallic Fe-Co) nanoparticles were synthesized using *Mentha piperita leaf* extract as reducing agent and Fe-Co as metal precusors. The synthesized NPs were characterized using UV-Visible FTIR, XRD and SEM analysis. The UV-visible results show their highest peak at 400 due to the surface plasmon vibration of the phytochemical constituent present in the extract. FTIR results show the presence of Alkaloids and triterpenes, SEM revealed the mono dispersed spherical and hexagonal shape of various sizes that are agglomated in nature. The XRD analysis shows the Face Cubic Centered Structure with the average crystalline size of 52.64 nm. Also significant antibacterial and antifungal activity when tested against *eschemicha-coli psendomonasaureginos* (Gram-negative), *staphylococcus aureus, Klebsella pneumonia* (Gram-positive), *Candida* and *Aspagillus Niger* (fungi) both showed increase inhibition growth with increase in concentrations

Compliance with ethical standards

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Disclosure of conflict of interest

Authors of this research work does not declare any conflict of interest regarding the draft of this research

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