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Article

Green Synthesis and Characterization of Cobalt, Iron and Copper Nanoparticles Derived from the Stem-Bark Extract of Khaya Senegalensis (Mahogany) and Its Antimirobial Activity

Nasiru Yahaya Pindiga [1,*], Khadija Aminu Ya'u [1], Adamu Abubakar [1], Yakong David Madugu [1]

[1] Department of Chemistry, Faculty of Science, Gombe State University Gombe, Nigeria

*Correspondence: Nasiru Yahaya Pindiga(npy500@yahoo.com)

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Abstract: During the past few decades, many of the synthetic chemicals are able to produce nanoparticles and nanoclusters, although these chemicals primarily act as reducing and capping agents, they are very toxic and hazardous and make the nanoparticles biologically incompatible. Thus there is need for green chemistry that includes a clean, non-toxic and environmental friendly method of nanoparticles synthesis. Cobalt, iron and copper nanoparticles were synthesized using the stem-bark extract of khayasenegalensis (mahogany) where cobalt chloride ($CoCl_2$ $6H_2O$), ferric chloride ($FeCl_2$), and copper sulphate ($CuSO_4$ H_2O) were used as the metal precursor respectively. The change in color from light brown to dark brown indicates the formation of cobalt nanoparticles, from light brown to dark green indicates the formation of copper nanoparticles and also the change in color from light brown to a dark color indicates formation of iron nanoparticles. The nanoparticles were further characterized using UV visible spectroscopy, FTIR, and SEM. The UV result for CoNPs showed the highest peak at 500nm and both FeNPs and CuNPs showed the highest peak at 300nm. The FTIR results for all the nanoparticles showed the presence of Alkaloids and triterpenes. Also the SEM result showed spherical granular, partially dispersed and monodispersed morphology for CoNPs, FeNPs and CuNPs respectively. Moreover, the antibacterial activity of the synthesized NPs when tested against two gram positive bacteria and two gram negative bacteria was evaluated and good results were obtained. The antifungal activity when tested against two fungi showed a very good result.

Keywords: Reducing agent, Khaya senegalensis, Nanoparticles

1. Introduction

Pollution control is one of the most important priority of the chemical industry worldwide. Green chemistry, also called sustainable chemistry, is an area of chemistry and chemical engineering focused on the design of products and processes that minimize or eliminate the use and generation of hazardous substances [1].

Primarily, green chemistry is characterized as a reduction of the environmental damage accompanied by production of materials and respective minimization and proper disposal of wastes generated during different chemical processes. According to another definition, green chemistry is a new technique devoted to synthesis, processing, and application of chemical materials in such manner as to minimize hazard to mankind and the environment. Numerous new terms have been introduced associated with the concept of "green chemistry", such as "eco-efficiency", "sustainable chemistry," "atom efficiency or atom economy", "process intensification and integration", "inherent safety", "renewable energy sources" etc [2]. The multidisciplinary nature of Green Chemistry is recognized worldwide as a route to the development of chemical products and processes with lower environmental impact. Green chemistry and sustainability have had a profound effect on the way industry wishes to be perceived. To promote uptake of Green and sustainable methodologies amongst the chemical and chemical-using industries requires the exemplification of green chemistry in education and training material to influence and inspire the next generation of scientists [3]. The development of efficient green chemistry methods for synthesis of nanoparticles has become a major area for research. There are a lot of investigations to find an eco-friendly technique for production of well characterized nanoparticles [4].

2. Materials and Methods

2.1. Reagents/ Chemicals

All the reagents used were of analytical grades and were as follows: $CoCl_2$, $FeCl_3$, $CuSO_4$, $5H_2O$, 1% tetraphynyl-p-phenylene diamine dihydrochloride. Ehrlich reagent (p-dimethyl amino benzaldehyde, absolute alcohol, hydrochloric acid), hydrogen peroxide, miller Hilton broth, nutrient agar, potato dextrose agar, sterile cotton, double distilled water, dimethyl sulphur-oxide, dimethyl dihydrochloride, crystal violet, iodine, acetone, ethanol, oil immersion, human plasma, griseofulvin, Gentamycin, and *khaya senegalensis* (mahogany) stem-bark.

2.2. Apparatus/ Equipment

The apparatus used are magnetic stirrer, heating mantle, beakers, volumetric flask, stirring rod, No. 1 whatman filter paper, FTIR machine, (Perkinelmer) spectrum version 10 03 09, UV-visible spectrophotometry machine, (Jenway 6705) SEM machine (Hitachi 4160), incubator, staining rags, autoclave, microscope, wooden mortar and pestle, swap stick and petri dish

2.3. Sample Collection

The sample-*khayasenegalensis* stem bark was collected at Gombe state university campus near the convocation square in Gombe state.

2.3.1. Sample Preparation

The *Khaya-senegalensis* (mahogany) stem bark collected was washed three times with tap water and then rinsed with distilled water. It was shade dried and grinded finely with the aid of a wooden mortar and pestle. It was then stored for further.

2.3.4. Extract Preparation

30g of the grinded sample was measured in a 500ml beaker and 200ml of distilled water was added to it. It was heated on a hot plate at 80^0C for 30minutes. It was allowed to cool and filtered using whatman No 1 filter paper. The filtrate was used immediately for the synthesis.

2.4. Preparation of the Metals Precursors

2.4.1. preparation of Cobalt Chloride Solution

A 0.01M $CoCl_2$ solution was prepared by suspending 2.3793g in a 1000ml volumetric flask and was filled up to mark using distilled water.

2.4.2. Preparation of Ferric Chloride Solution

0.01M solution of $FeCl_3$ was prepared by dissolving 1.6231g of $FeCl_3$ salt into 1000ml volumetric flask, and was filled up to the mark with distilled water.

2.4.3. Preparation of Copper Sulfate Solution

0.01M $CuSO_4$ .5H20 Solution was prepared by dissolving 2.49g of $CuSO_4$ .5H20 in 1000ml volumetric flask and was filled up to the mark with distilled water.

2.5. Synthesis of Copper Nanoparticles

By using slightly modified method of [5]. The prepared $CuSO_4.5H_2O$ solution was added drop wise into the plant extract in a ratio of 2:8 (that is 20ml plant extract and 80ml metal precursor) with constant stirring at 80^oC for 60 minutes using magnetic stirrer. Within the first 30 minutes, color change was observed which indicated the formation of nanoparticles. It was then kept and allowed to settle for 24 hours. After which it was decanted and dried. The collected nanoparticles at 100^oC for 2hours and were ground into powdered and kept for further analysis.

2.6. Synthesis of Iron Nanoparticles

By using slightly modified method of [6] For the synthesis of iron nanoparticles, the prepared $FeCl_3$ solution was added drop wise into the plant extract in a ratio of 1:5 (that is 10ml extract and 50ml metal precursor) with constant stirring at 60^0C for 60 minutes using magnetic stirrer. Within the first 15 minutes, the color was observed which indicated the formation of nanoparticles. It was then kept and allowed to settle for 24hours after which it was decanted and dried. The collected nanoparticles at 100^oC for 2hours was then ground into powdered for further analysis.

2.7. Synthesis of Cobalt Nanoparticles

During synthesis of CoNPs (Cobalt nanoparticles), 15 mL of whole plant extract was added dropwise to 50 mL of 0.003 M aqueous solution of $CoCl_2$ with constant stirring at 80 °C. Change in color of the reaction mixture indicated the formation of cobalt nanoparticles [7], It was then kept and allowed to settle for 24 hours. After which it was decanted and dried. The collected nanoparticles at 100^oC for 2hours and were ground into powdered and kept for further analysis.

2.8. Characterization

2.8.1. UV- Visible Spectroscopy

UV-Visible spectroscopy analysis was done to confirm the formation of nanoparticles and observed the extract plasmon vibration and excitation. The wavelength was varied at regular wavelength of 200nm, 300nm, 400nm, 500nm, 600nm, 700nm and 800nm respectively. Maximum absorption wavelength was determined by placing each aliquot sample in quartz cuvette, using distilled water as the blank or reference solvent.

2.8.2. Fourier Transform Infrared (FTIR)

FTIR analysis was done on the stem-bark extract and the synthesized Cobalt, Iron and Copper nanoparticles to determine the functional group present in the samples. The infrared spectra measurements were carried out using a Perkin Elmer spectrophotometer model 10.03.09 at the university Pharmaceutical department Gombe State University, Gombe State to identify the possible phytochemicals responsible for the reduction, capping and stabilization of metal NPs.

2.8.3. Scanning Electron Microscopy (SEM)

The scanning electron microscopy was carried out in order to determine the morphology of the element present in the synthesized Cobalt, Iron and Copper nanoparticles. It was done using powdered nanoparticles obtained during the synthesis.

2.9. Antimicrobial Activity

2.9.1. Media Preparation

The media preparation depends on the manufacturer's specification. 28g of nutrient agar was dissolved into 1000ml distilled water and autoclaved at 121°C for 15 minutes to sterile.

2.9.2. Bioactivity Studies

The antimicrobial activity studies were carried out by using the agar well method to test the antimicrobial activity of the green synthesized cobalt, iron and copper nanoparticles on two gram-negative bacteria's *Escherichia-coli, pseudomonas aeruginosa*. Two gram-positive bacteria; *Staphylococcus aurous, Salmonella typhi* and two fungus; *A. Nigerand Candida albicans* as well. This will be conducted by creating 6mm hole in the prepared agar (media) inside the petri dish. The organism was inoculated all over the surface of the petri dish and the synthesized drug (nanoparticles) was also inoculated in to each hole with a control drug at the center. It was then incubated overnight at 37°C after which the zone of bacteria and fungi growth inhibition was measured in millimeters (mm).

3. Results

3.1. Characterization

3.1.1. UV Visible Spectroscopy

The cobalt nanoparticles showed the highest absorbance at peak 500nm (Figure 1) which is due to the surface plasmon vibration and excitation of the bio reduction and capping agent present in the stem-bark extract which correspond to the result obtained by [8] that showed maximum adsorption peak at 448nm. It also tallies fairly with the result of [9] that showed maximum absorption peak at 438nm when cobalt nanoparticles were green synthesized using Asparagus racemosus root Extract.

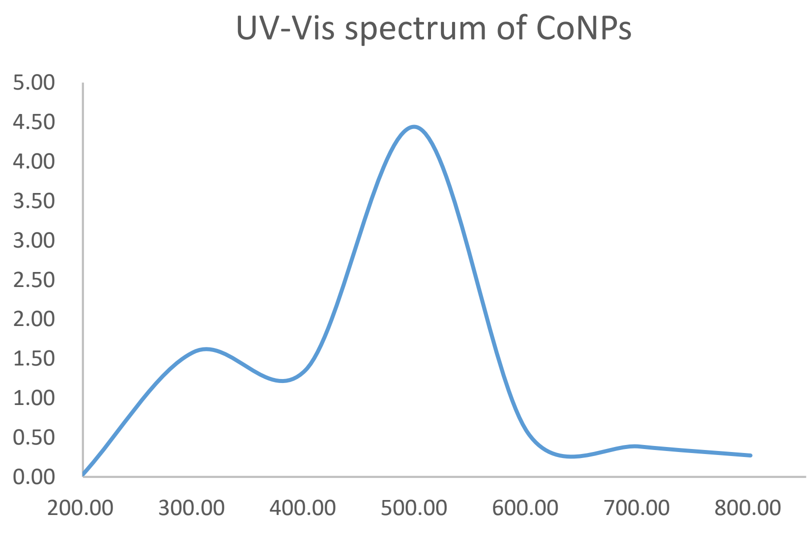

Figure 1. UV-Visible spectroscopy result of CoNPs

The iron nanoparticles showed the highest absorbance at peak 300nm (Figure 2) which is due to the surface plasmon vibration and excitation of the bio reduction and capping agent present in the leaf extract. This corresponds to that obtained by [10] which showed the highest absorbance peaks at 350,315, and 400nm. They said, various report has been established that the absorption peaks of iron nanoparticles (FeNPs) appears should be around 280-420nm.

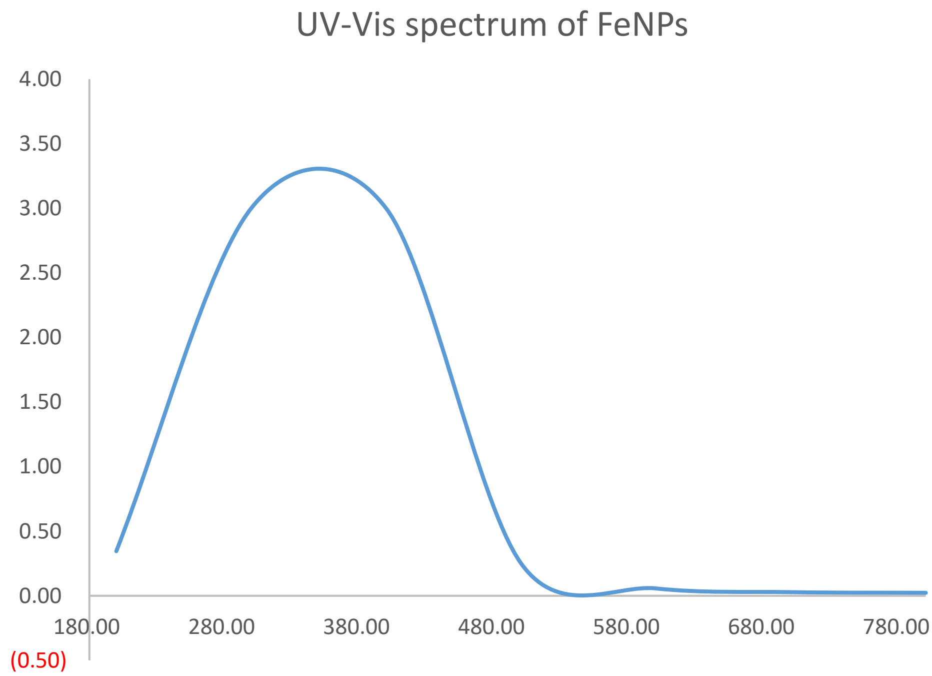

Figure 2. UV-Visible spectroscopy result of FeNPs

The copper nanoparticles showed the highest absorbance at peak 300nm (Figure 3) which is due to the surface plasmon vibration and excitation of the bio reduction and capping agent present in the stem extract which correspond to the result obtained by [11] in which the highest peak is at 340nm when synthesized using *Cissus vitiginea*.

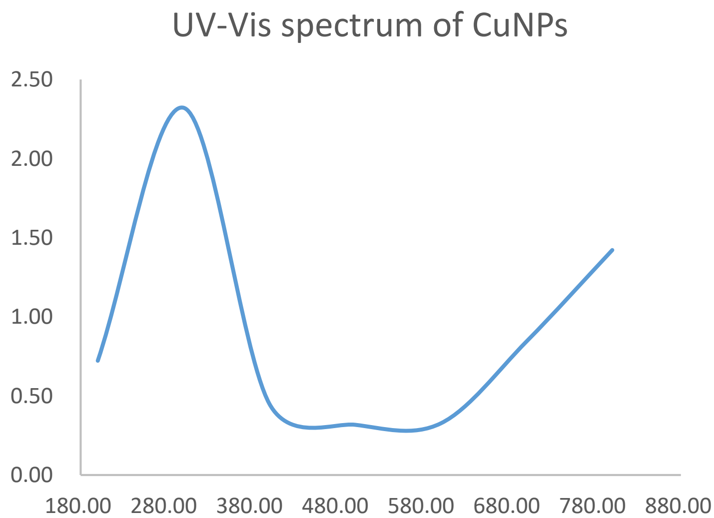

Figure 3. UV-Visible spectroscopy result of CuNPs

3.1.2. Fourier Transform Infrared Spectroscopy

The FTIR results for the green synthesized nanoparticles (Figure 4) showing different functional groups were discussed below

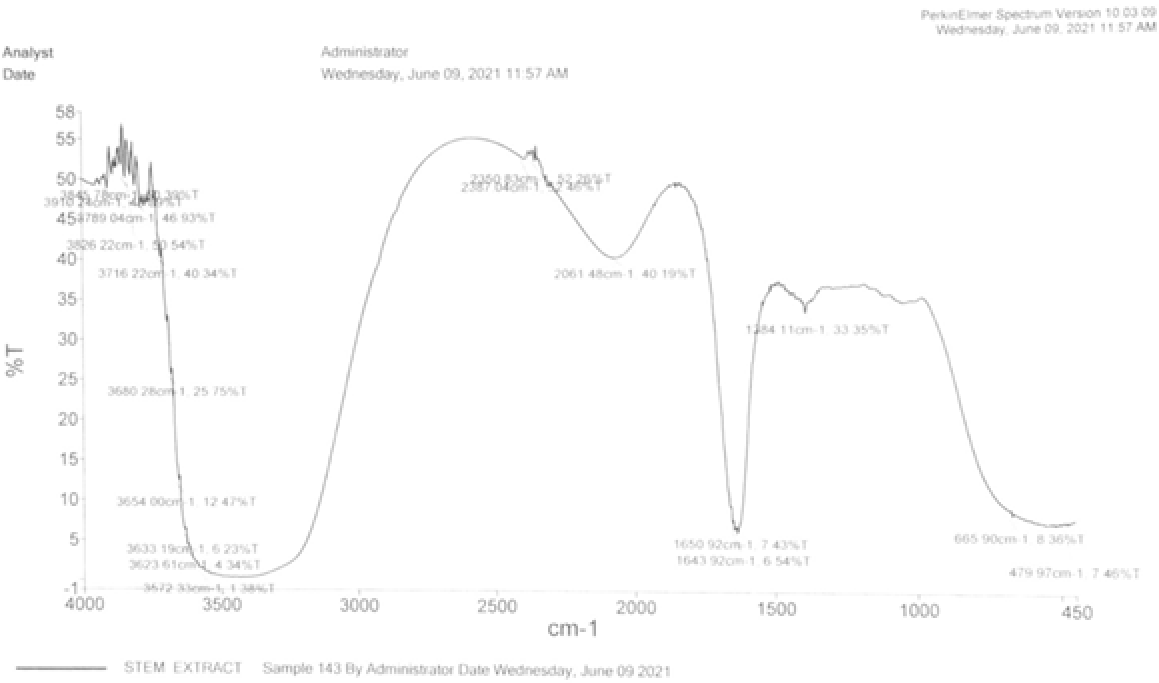

Figure 4. FTIR result of stem-bark extract

The FTIR spectra of the stem extract (Figure 4) shows prominent peaks at 2350.92cm^{-1}, 2061.48cm^{-1} , 1650.92cm^{-1}and 3572.33cm^{-1}. The peak at 1650.92cm^{-1} indicates C=N stretching which is due to the triterpenes, the absorption at 3572.33cm^{-1} indicates O-H bond due to the presence of water.These correspond to the literature obtained by [12].

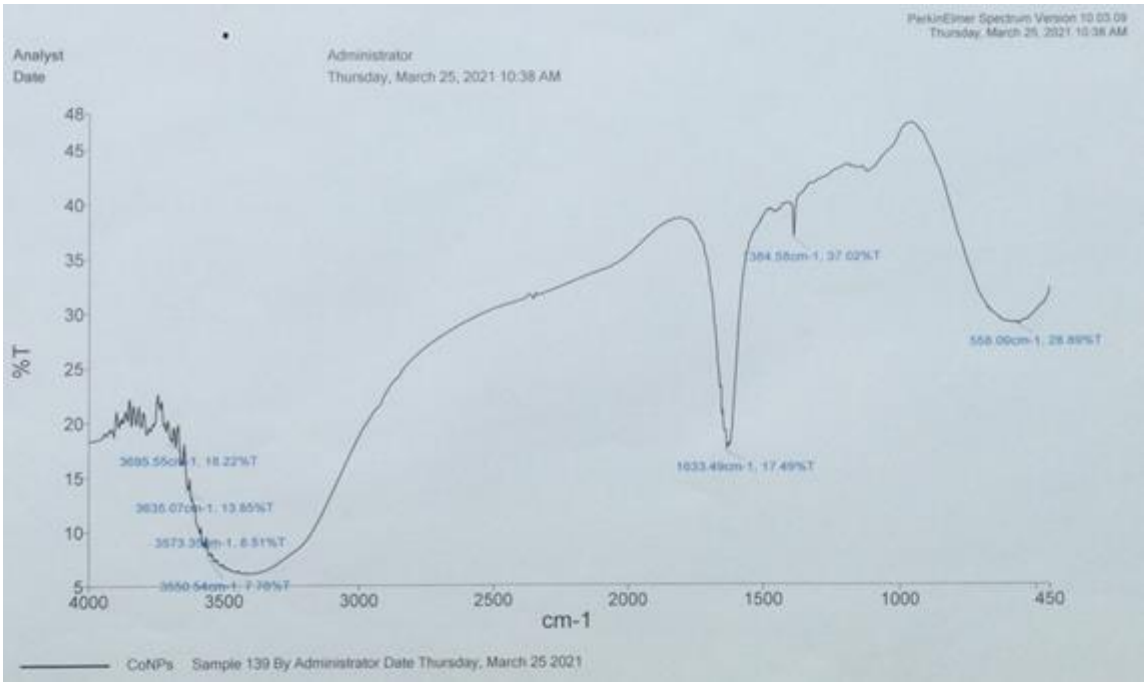

Figure 5. FTIR result of CoNPs

The FTIR analysis of CoNPs (Figure 5) shows peak at 3695.55cm-^{1}, 3635.55cm^{-1} 1633.49cm^{-1}, 1384.58cm^{-1} and 558.09cm^{-1}. The 4 prominent peak at 3695.55cm-^{1}, 3635.55cm^{-1}, are due to O-H stretch which indicates the presence of alcohol or phenol. The adsorption at 1633.49cm^{-1} indicate the C=C due to alkene which is due to the linkage of metal and extract, band at 1384.58cm^{-1} is due to asymmetric stretching of COO and band at 558.09cm^{-1} is due impurities which correspond appreciably with the result by [9].

These have replaced those observed in the spectrum of the stem extract that include peaks at 2350.92cm^{-1}, 2061.48cm^{-1}, 1650.92cm^{-1}and 3572.33cm^{-1}. Most noticeable appearance was disappearance of peaks at 2350.92cm^{-1}, 2061.48cm^{-1} and 3572.33cm^{-1}. The broadening of the bands took place in presence of NPs which was the result of the interaction between extract components and NPs either by binding with the functional groups or capping them [13].

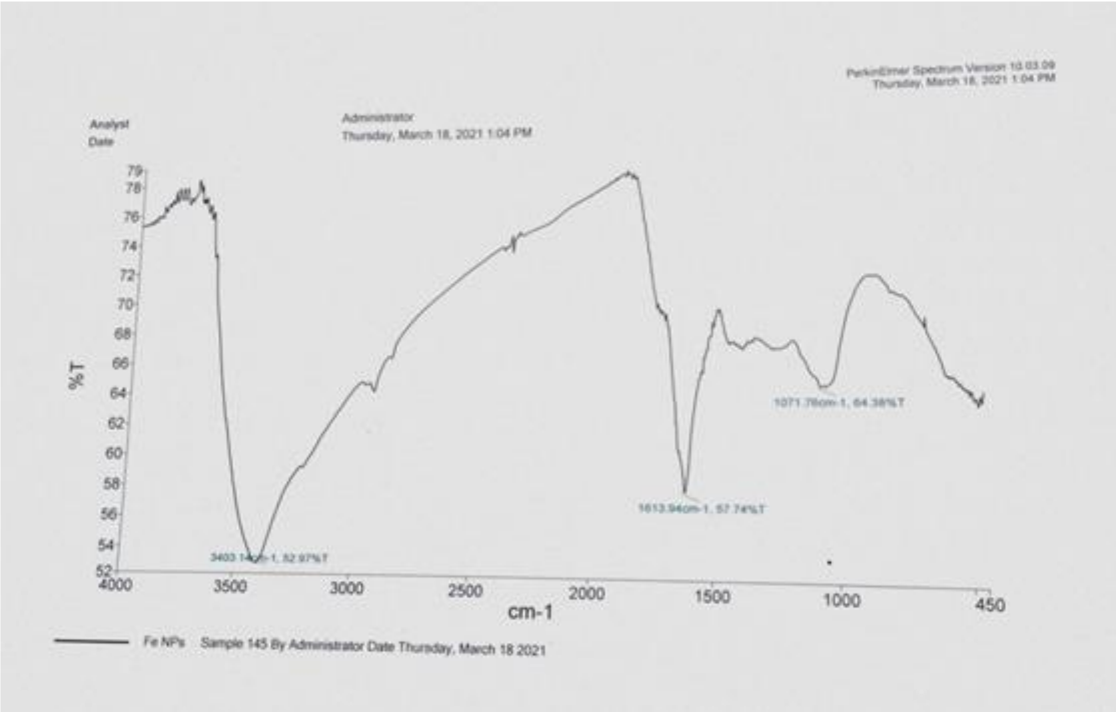

Figure 6. FTIR result of FeNPs

The FTIR analysis of FeNPs (Figure 6) shows peaks at 3403.14cm^{-1}, 1613.94cm^{-1}, 1071.76cm^{-1}, and 450cm^{-1}. The peak at 3403.14cm^{-1} indicates the N-H stretch due to primary and secondary amines or amides in the alkaloids due to the phytochemical constituent present in the extract. Absorption band at 1613.94cm^{-1} indicates C-C stretch in the ring due to aromatic ring in the alkaloids and terpenes. Band at 1071.76cm^{-1} =C-H indicates bend due to alkene present in the ring and that of 450cm^{-1}. This result corresponds with the literature of [14]. These have replaced those observed in the spectrum of the stem bark extract that include peaks at 2350.92cm^{-1}, 2061.48cm^{-1}, 1650.92cm^{-1}and 3572.33cm^{-1}. Most noticeable appearance was peak at 1071.76cm^{-1}and the disappearance of peaks at 2350.92cm^{-1}, 2061.48cm^{-1}and 3572.33cm^{-1}.

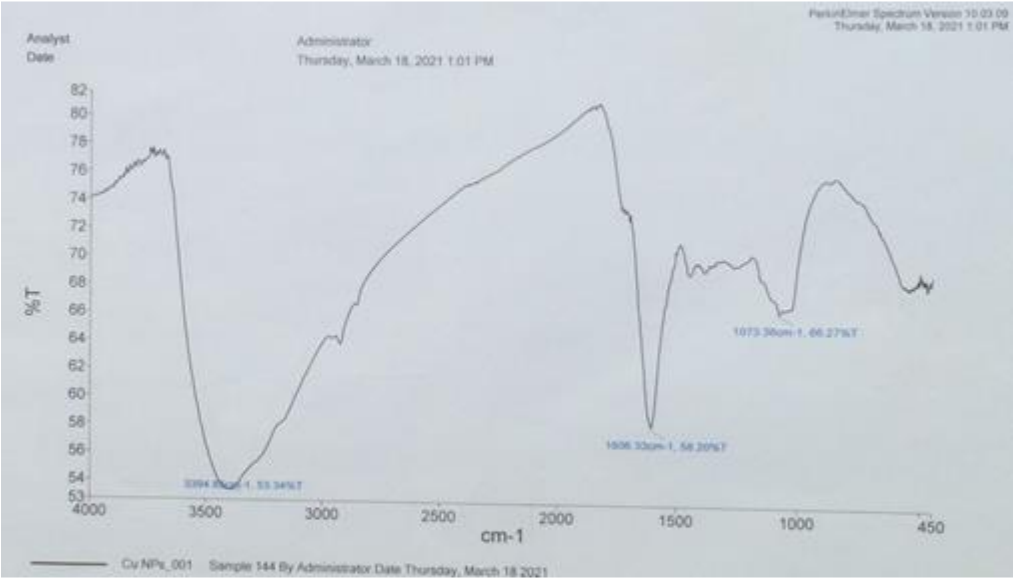

Figure 7. FTIR result of CuNPs

The FIIR result of CuNPs (Figure 7) shows absorption peak at 3394.85cm^{-1}, 1606.33cm^{-1}, 1073.38cm^{-1}. Peak at 3394.85cm^{-1} was due to O-H bond of the alcohol, carboxylic acid or phenol in the phytochemical and band at 1606.33cm^{-1} arise C=O of COOH, respectively. The very strong band at 1072 cm-1 arises from C-O-C symmetric stretching and C-O-H bending vibrations of protein in the *khayasenegalensis.* This result corresponds fairly with that obtained by [14].

3.1.3. Scanning Electron Microscopy

The Scanning electron microscopic analysis (Figures 8, 9 and 10) was employed to identify the morphologies (shape) of the synthesized nanoparticles

The result of the SEM analysis of cobalt nanoparticles (Figure 8) showed a spherical granular morphology.

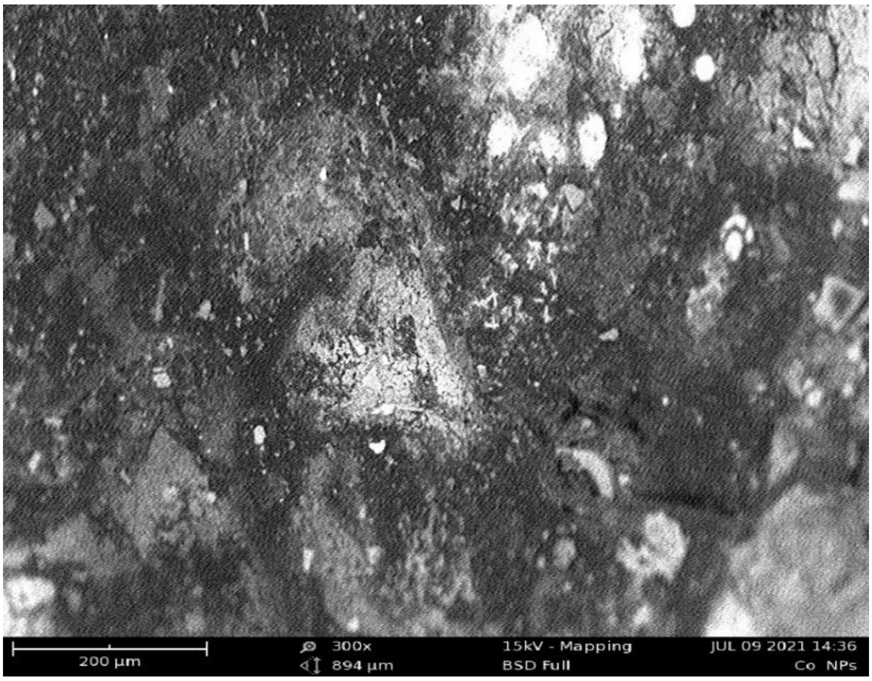

Figure 8. SEM image of CoNPs

Iron nanoparticles (Figure 9) showed a partially dispersed spherical morphology.

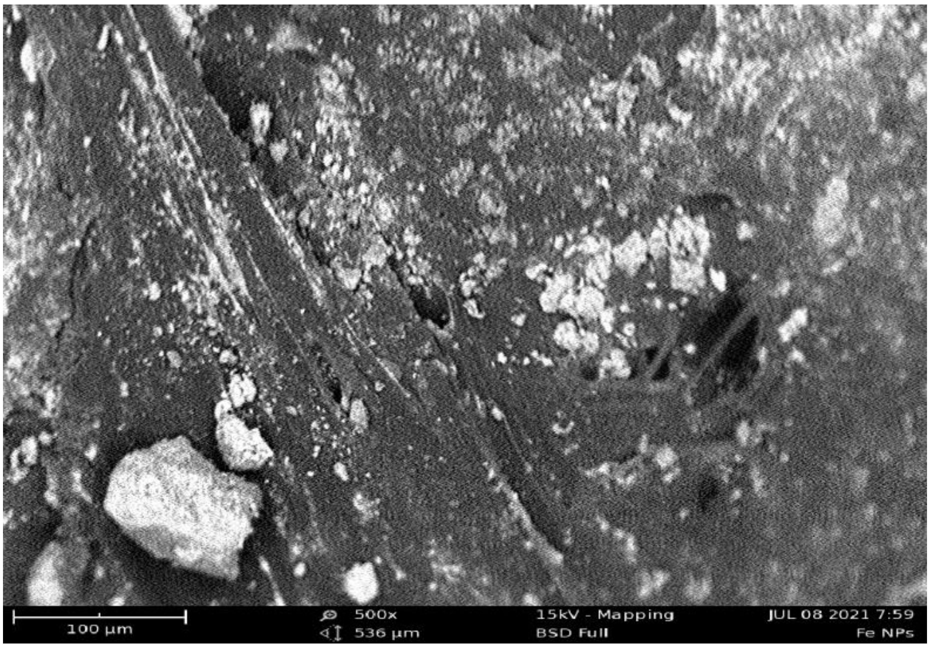

Figure 9. SEM image of FeNPs

The SEM analysis of CuNPs (Figure 100) showed a monodispersed and agglomerated morphology.

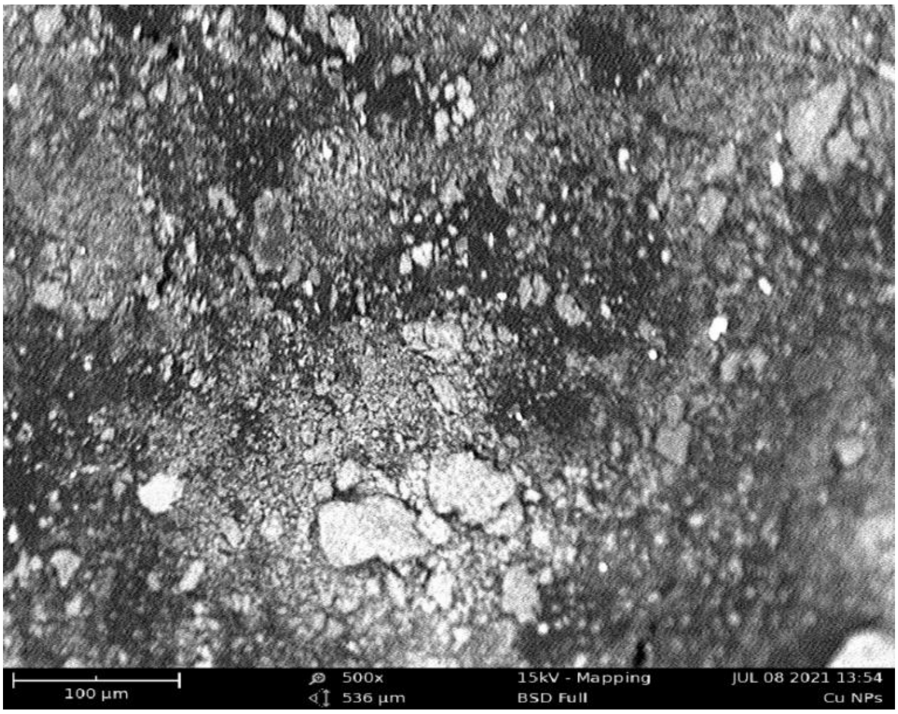

Figure 10. SEM image of CuNPs

3.4. Antimicrobial Study

3.4.1. Antibacterial Assays

The antibacterial activity of the synthesized nanoparticles was conducted against some selected bacteria were shown below; (Figure 111 – 16).

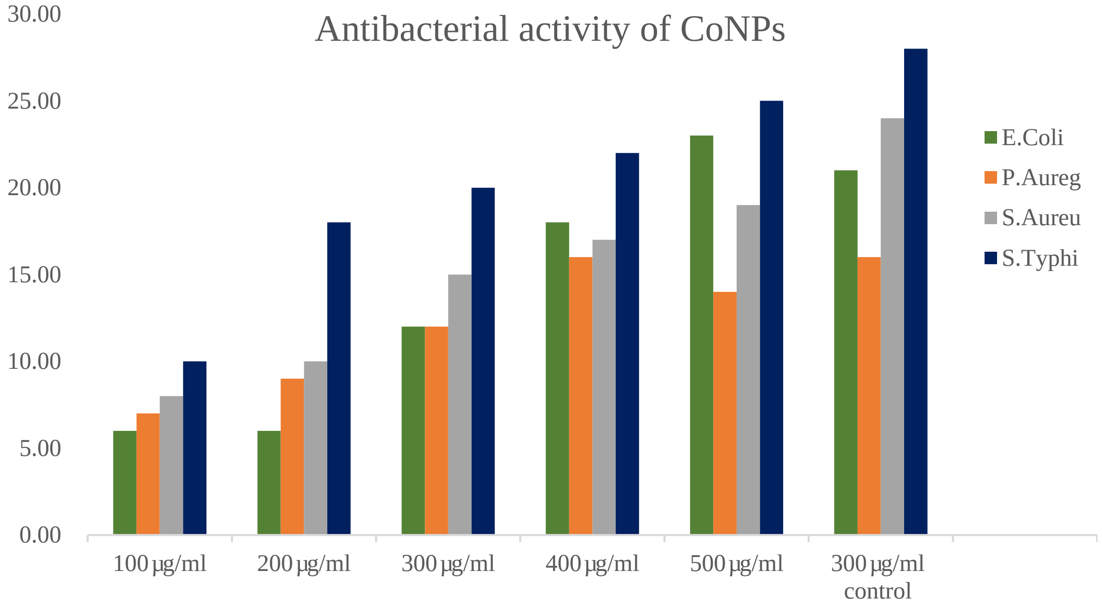

Figure 11. Antibacterial activity of CoNPs; Control = Gentamycin, *E. Coli* -= Escherichia Coli, *P. Aureg* = Pseudomonas Aureginosa, *S. Aureu* = Staphyloccocus Aureus, *S-Typhi* = *Salmonella Typhi*, µg/ml = Microgram per mil

Note, each result was obtained by taking the average mean in (mm) where n=2,6mm in indicate no activity i.e it only resist the drug, 9mm shows weak resistance, 16mm shows moderate resistance and 16 above are significant resistance.

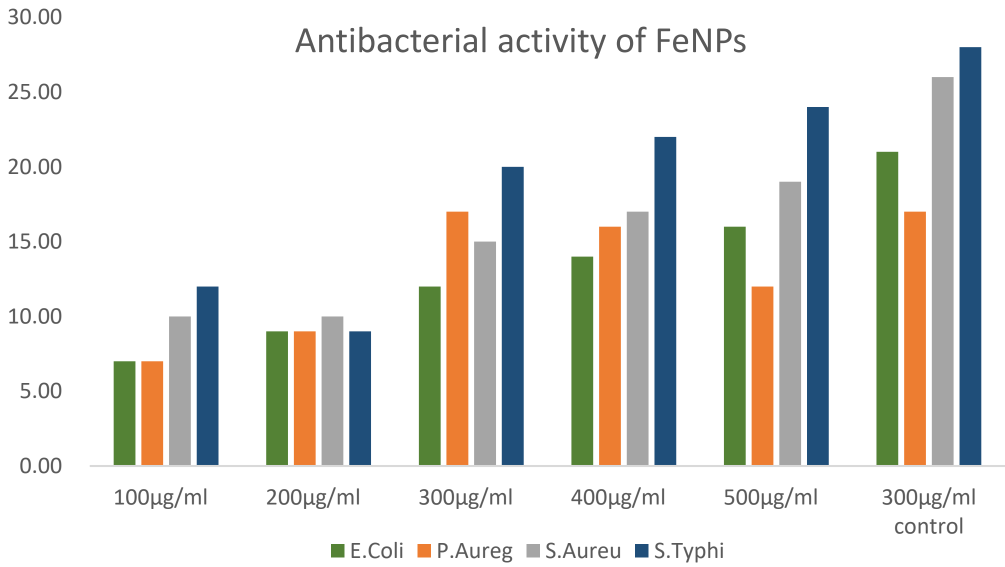

Figure 12. Antibacterial activity of FeNPs; Control = Gentamycin, *E. Coli* -= Escherichia Coli, *P. Aureg* = Pseudomonas Aureginosa, *S.Aureu* = Staphyloccocus Aureus, *S-Typhi=Salmonella Typhi*, µg/ml = Microgram per mil

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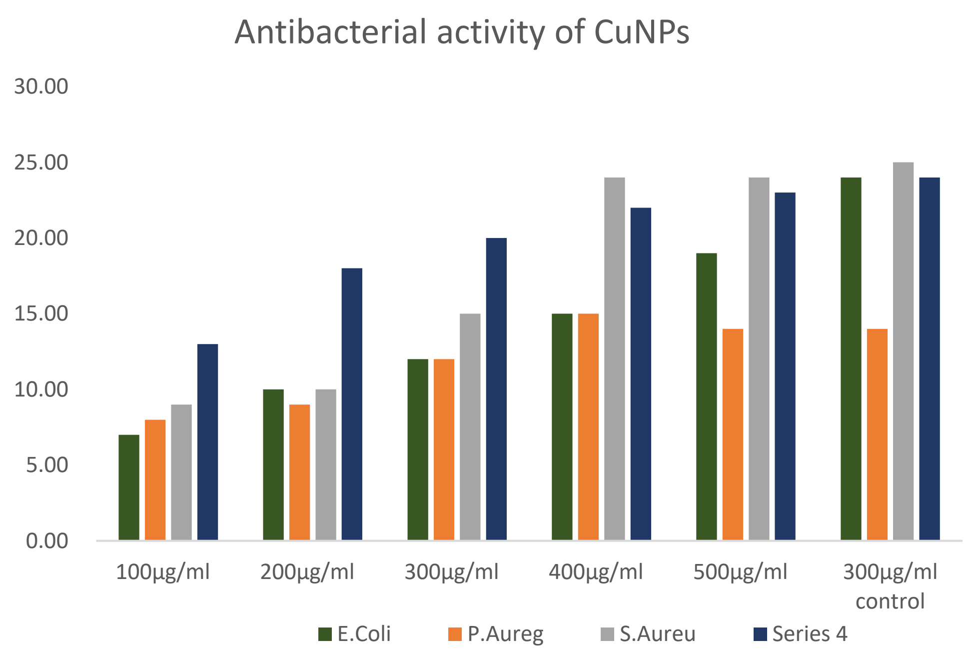

Figure 13. Antibacterial activity of CuNPs; Control = Gentamycin, *E. Coli* -= Escherichia Coli, *P. Aureg* = Pseudomonas Aureginosa, *S.Aureu* = Staphyloccocus Aureus, *S-Typhi=Salmonella Typhi*, µg/ml = Microgram per mil

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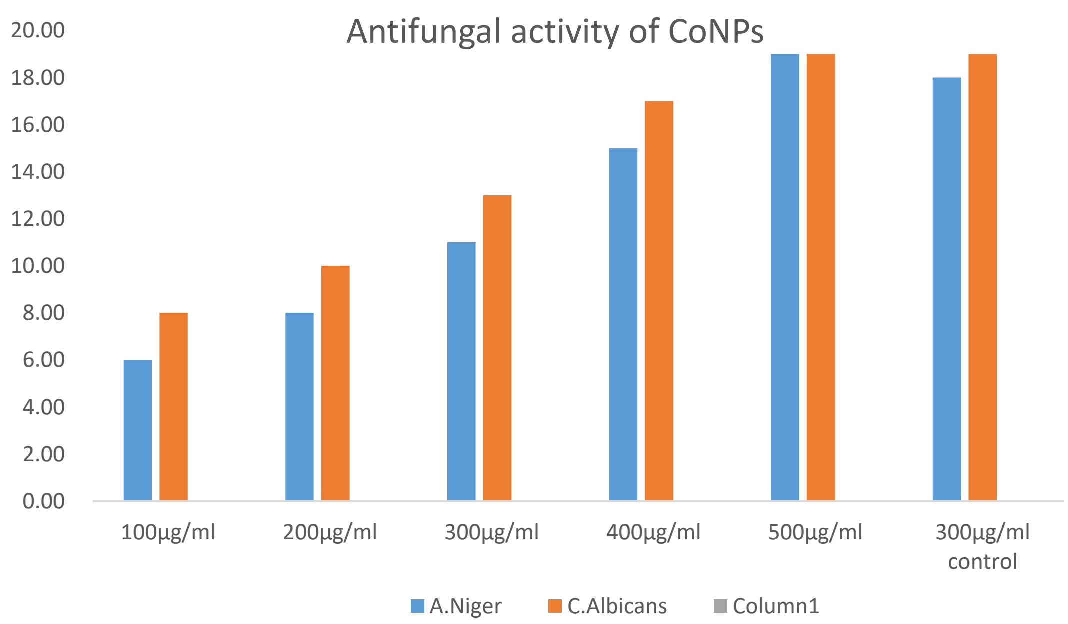

Figure 14. Antifungal activity of CoNPs; Control = Fulcin, *A.Niger* = Aspagillus Niger, *Candida* = Fungus Candida, µg/ml = Microgram per mil

Note, each result was obtained by taking the average mean in (mm) where n=2,6mm in indicate no activity i.e it only resist the drug, 9mm shows weak resistance, 16mm shows moderate resistance and 16 above are significant resistance.

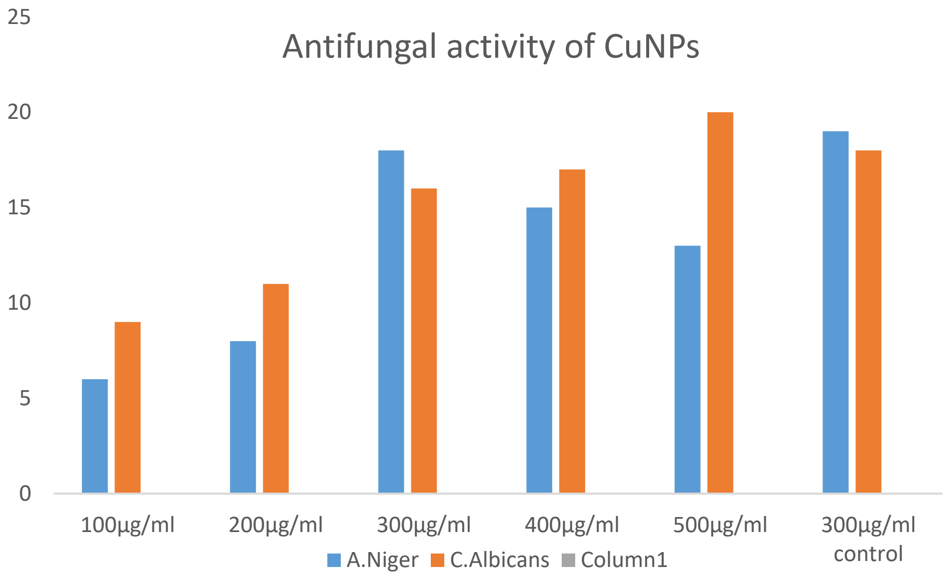

Figure 15. Antifungal activity of CuNPs; Control = Fulcin, *A.Niger* = Aspagillus Niger, *Candida* = Fungus Candida, µg/ml = Microgram per mil

Note, each result was obtained by taking the average mean in (mm) where n=2,6mm in indicateno activity i.e it only resist the drug, 9mm shows weak resistance, 16mm shows moderate resistance and 16 above are significant resistance.

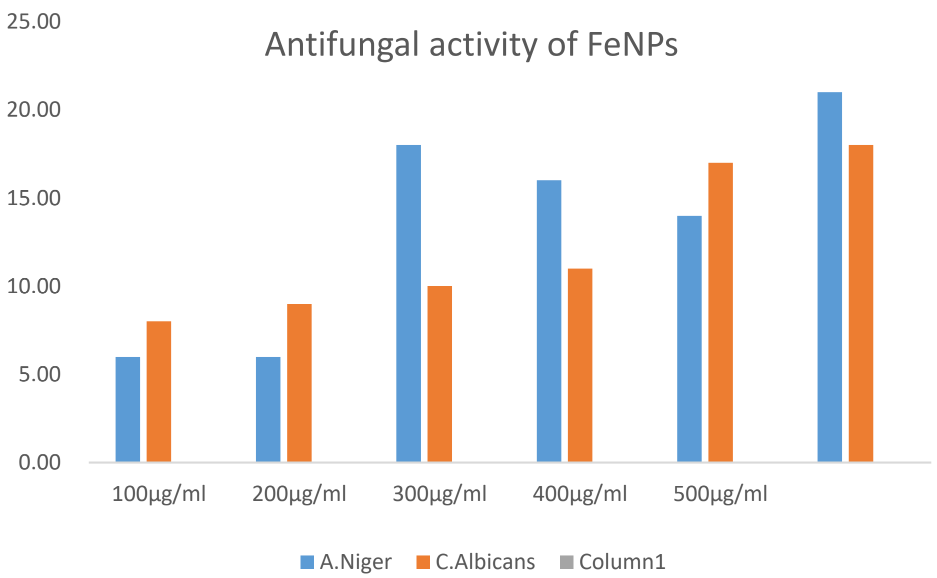

Figure 16. Antifungal activity of FeNPs; Control = Fulcin, *A.Niger* = Aspagillus Niger, *Candida* = Fungus Candida, µg/ml = Microgram per mil

Note, each result was obtained by taking the average mean in (mm) where n=2,6mm in indicate no activity i.e it only resist the drug, 9mm shows weak resistance, 16mm shows moderate resistance and 16 above are significant resistance

4. Conclusions

The Cobalt, Iron and Copper nanoparticles were green synthesized from the stem-bark extract of *khayasenegalensis* (mahogany) with the aid of non-toxic and inexpensive chemicals and characterized using UV visible spectrometry, Fourier Transform Infrared Spectrometry and Scanning Electron Microscopic analysis. The synthesized nanoparticles also showed good antibacterial and antifungal activity against some selected bacteria and fungi.This analysis suggests that, synthesized nanoparticles the stem- bark of *KhayaSenegalensis* contain active agent(s) and this provides the basis for their traditional-medicinal use as a cure for some human ailments. This assertion is also confirmed, as their extracts indicate a relatively moderate number of phytochemicals present.

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