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Synthesis Of Copper Nanoparticles From E-Waste , Characterization By Tem And Uv-Vis Spectrophotometry To Evaluate Their Antioxidant Application

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Abstract
The green synthesis of copper nanoparticles (Cu NPs) using a leaf extract from Parthenium hysterophorus has been documented in our present research work. The existence of flavonoids, tannins, glycosides, and alkaloids was confirmed by the phytochemical analysis of the plant extract and these chemicals can be used as reducing, stabilizing and capping agents. The Cu NPs were characterised by TEM and UV-vis spectrophotometry. The average sizes of the nanoparticles are found between 5-50nm indicating the effective reducing activity of plant extract and has many environmental applications.
Keywords: *Nanoparticles, Phytochemical analysis and Ultraviolet spectrophotometry.*

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I. Introduction

Exponential growth in the electrical and electronic industries to meet customer demand has correspondingly generated large waste flows. Electronic and electrical waste (e-waste) can be defined as any "electrical or electronic components, that has reached the end of its usable life cycle, including all components, subassemblies and consumables, which are part of the equipment at the time the equipment becomes waste". E-waste contains a number of hazardous constituents that have a negative impact on the environment and, more importantly, human health if not properly managed. Electronics actually contain toxic substances - therefore they must be handled with care when no longer wanted or needed. Instead of just being thrown in the garbage, it must be thrown away by a certified e-waste hauler or recycler, or taken to a designated drop-off at a government building, school or organization as e-waste can potentially cause harm to humans, animals and the global environment if disposed of improperly. As a result of its harmful influence on human health, it poses a hazard to the ecosystem.

During the last two decades, the adsorption process has been widely utilized in the field of environmental protection. A wide variety of adsorbents like activated carbons have been developed and tried to expel various toxins from aqueous solution[1]. Recently, explorers have discovered new materials with high- capacity adsorbent properties to toxic remediation pollutants from water. General applications of copper oxide include biomedical (antimicrobial, anti-fouling, antifungal, antibiotics, antioxidants, drug delivery, and anticancer), textile industries, thermo sensing and conducting materials, gas sensors, catalytic, synthesis of inorganic-organic nanosized composites, magneto resistant materials, high-temperature superconductor, environmental remediation, etc. a biological method is also followed for the green chemistry synthesis of metal nanoparticles due to it being free from hazardous chemicals. The metal nanoparticle synthesis method from plant extracts has more advantages over the microbial synthesis method because the microbial process is highly expensive due to the cost of microorganism isolation and their culture maintenance. Fe, Cu, Zn and Ag nanoparticles have been synthesized using biological sources and have been reported in potential applications such as for antimicrobials, photocatalytic activity *etc.* Similar leaching methods has been reported by Aziz Mikaeli Hamzeh Ghasem and Shahrzad Khoramnejadian [2015], extracted 99.92 % gold from E-waste using a hydrometallurgy method.

The multifunctional activity of green-synthesized nanoparticles using e-waste is not reported in any of the previous research, we focused on the multifunctional activity of Cu NPs. Green Synthesis of copper nanoparticles from e- waste is being studied in my work as Cu nanoparticles have unique properties. E-waste minimization and proper disposal is taken into consideration. The weed plant parthenium is taken for study as it has active secondary metabolites to produce nano particles. Our research focussed on synthesis of copper nanoparticles from E-waste using Parthenium plant. **Huan Li and Elsayed Oraby [2022],** developed a greener method for extract precious metals from waste printed circuit boards (PCB) using alkaline glycine solution in the presence of an oxidant.

Experiment

Parthenium hysterophorus plant grown in empty lands in Nagarjuna university campus, Nagarjuna Nagar, Guntur, AP was collected and washed thoroughly and washed with distilled water. The dried *Parthenium hysterophorus* plant material was powdered chopped into fine pieces. About 1.0 gram of dried plant material taken in 50 ml beaker and 100 mL of double distilled water added. This mixture was boiled at 60°C in the water bath for one hour. The solution was cooled at room temperature and filtered by Whatman filter paper No. 1. The filtrate was collected and stored at 4°C for further experimentation.

The Parthenium hysterophorus plant collected for green synthesis was identified by its main physiological features of leaves color and shape, flowers. The phytochemicals especially secondary metabolites that involves in reduction of Cu ions are determined by preliminary phytochemical analysis. The aqueous extract was tested for qualitative analysis of secondary metabolites. The results confirm the presence of alkaloids, flavonoids, phenolic compounds and terpenoids in aqueous plant extract.

Presence of alkaloids were tested with Hager's reagent and creamy white colour precipitate formation confirms the presence alkaloids. Orange coloured solution formation with sulphuric acid confirms the presence of flavonoids. Lead acetate give the white colour precipitate confirms the presence of phenolic compound. And appearance of grey colour solution while addition of chloroform and sulphuric acid confirms the presence the of terpenoids. Presence of different secondary metabolites confirms the plant is rich for different organic chemicals that can reduce the copper form the metallic salt solution for synthesis of copper oxide nanoparticles. Metal ions in solution interact with Flavonoids and polyphenolic compound and helps in the nucleation and reduction of $Cu^{2+}$from $Cu(NO_3)_2 \cdot 3H_2O$ to CuONPs [Nazar et al, 2018].

Figure 1: Parthenium hysterophorus Plant material collected for CuO NPs synthesis

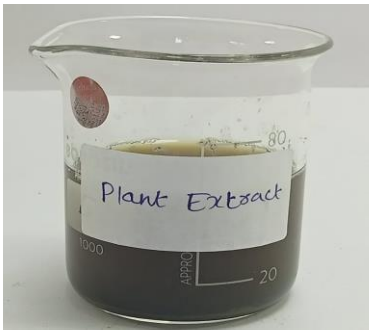


Figure 2: Aqueous extract of Parthenium hysterophorus Plant

Synthesis of copper oxide nanoparticles

The CuONPs were synthesized by using copper metallic salt derived from PCB as a precursor and *Parthenium hysterophorus* extract as a reducing and capping agent. The plant material preparation and extract are presented. After the addition of extract with $CuSO_4$ and metallic salt, the formation of CuONPs confirmed by the change in colour from blue to light brownish visually indicates the formation of copper oxide NPs. Change in colour of $CuSo_4$ solution and metal salt from PCB are presented. The nanoparticles were washed by distilled water and ethanol to remove any unwanted particles. Flavonoids present in the plant extract can precisely search for the active oxygen species (ROS) and have the ability to donate the hydrogen or electron, and the phenolics exhibit a chelating effect on the metal ions. Metal ions in solution interact with Flavonoids and polyphenolic compound and helps in the nucleation and reduction of $Cu^{2+}$from Cu $(NO_3)_2 \cdot 3H_2O$ to CuONPs [Nazar et al, 2018]. The interaction between conduction electrons of metal NPs and incident photons was responsible for colour change [Jana et al, 2017].

The presence of phenolic compounds, tannins, and different glycosides was confirmed during the phytochemical screening of *Parthenium hysterophorus* extract. The details of the screening are as given. It is possibly believed that the bioactive compounds such as alkaloids, flavonoids, phenolic compounds and terpenoids identified in the aqueous plant extract act as a ligand and bind to metal ions and reduce them and cap them to form nanoparticles. These ligands also act as particle size controllers as reported by the earlier researcher [Khatami et al [2017]. The dried CuoNPs are subjected to various characterization methods. A shiny black color of Cu-NPs was obtained after centrifugation and drying.

Parameter	Nanoparticles from $CuSo_4$ solution			Nanoparticles from PCB metal salt		
	0.1M	**0.01M**	**0.001M**	**0.1M**	**0.01M**	**0.001M**
Yield	**80 mg**	**110 mg**	**60 mg**	**75mg**	**92 mg**	**58 mg**

Table 1: Yield of green synthesized CuO NPs

Based on the yield of copper oxide nano-particles, Time for the formation and the spectrophotometer absorption results, 0.1M concentration of metal solution and *Parthenium hysterophorus* as a biological reducing agent was selected for further studies. The green synthesized copper nanoparticles were characterized with UV Visible spectroscopy and transmission electron microscopy (TEM).

UV spectral analysis

The UV-Vis spectra of copper oxide nanoparticles synthesized using a *Parthenium hysterophorus* extract are shown in figure. As it can be seen from spectra, peak of maximum absorption is exhibited between 260 to 290 nm which are attributed to the formation of copper oxide. An increase in the intensity of the peaks with the ratio decrease is observed. This may be due to the increasing number of nanoparticles formed as a result of copper ions reduction [**Ahmad and Sharma**, 2012], which may be caused by the complete enlacement of the copper ions by the extract. This is attributed to the oscillation of surface conduction electrons, which are excited by the incident electromagnetic radiation. The peaks represent the synthesis of CuONPs from standard copper sulphate solution (1.0M, 0.1 M and 0.01 M) respectively. All three concentrations exhibit the absorption maxima at 285nm, 270 nm and 265nm. All three concentrations exhibit the absorption maxima at 278nm, 270 nm and 264nm. The intensity of maximum absorbance peak was found high compared to the spectra of CuO NPs from PCB. The PCB derived metal salt consist of other metals in trace amount which may interrupt the reduction process, hence the intensity was found high in nanoparticles from copper sulphate solution. The *Parthenium hysterophorus* extract has successfully reduced the copper ions in both copper sulphate and metal salt solution for synthesis of CuO NPs.

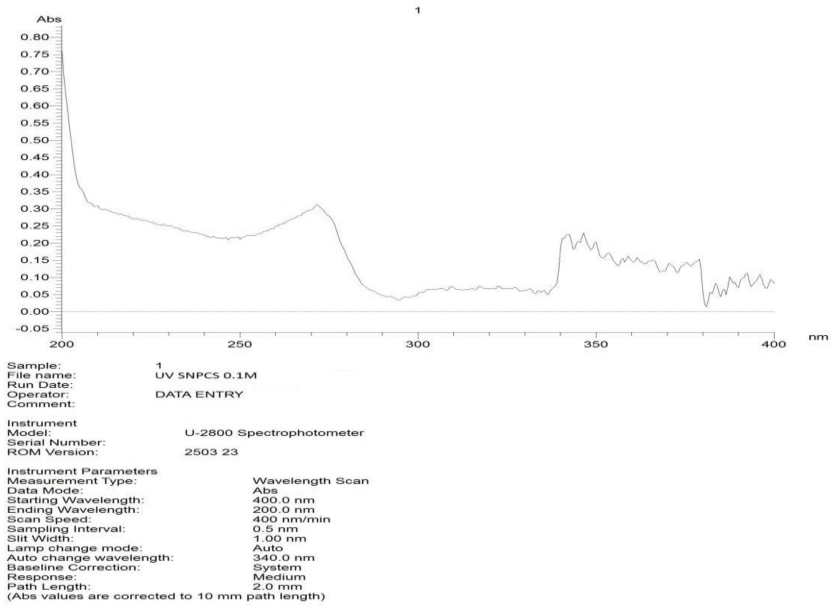
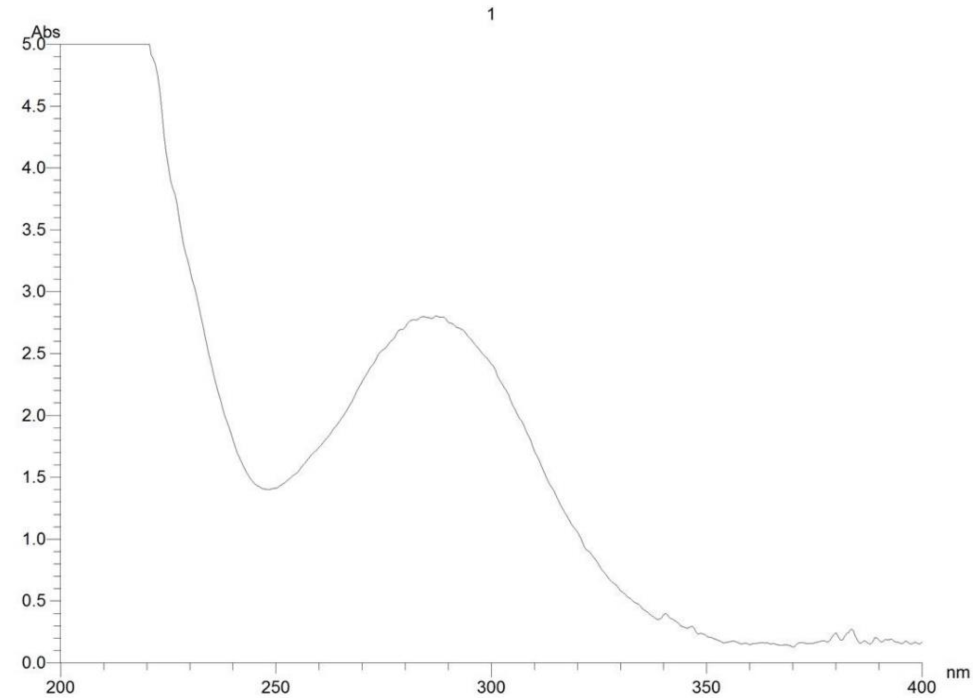


Figure 3: The UV-Vis spectra of CuONPs synthesized from copper sulphate (0.1M)



Sample: 1
File name: UV SNPPCB 0.1M
Run Date:
Operator: DATA ENTRY
Comment:

Instrument
Model: U-2800 Spectrophotometer
Serial Number:
ROM Version: 2503 23

Instrument Parameters
Measurement Type: Wavelength Scan
Data Mode: Abs
Starting Wavelength: 400.0 nm
Ending Wavelength: 200.0 nm
Scan Speed: 400 nm/min
Sampling Interval: 0.5 nm
Slit Width: 1.00 nm
Lamp change mode: Auto
Auto change wavelength: 340.0 nm
Baseline Correction: System
Response: Medium
Path Length: 2.0 mm
(Abs values are corrected to 10 mm path length)

Figure 4: The UV-Vis spectra of CuONPs synthesized from metal salt from PCB (0.1M)

TEM Analysis

This analysis is done to know the use of transmitted electrons, the electrons that are passing through sample before they are collected. To visualize the structural characteristics and topographical analysis, purified CuO NPs are examined under Transmission electron microscopy (TEM). The nano particles were stained and saturated aqueous urenyl acetate (UA) and Reynolds lead citrate (LC), respectively. The sections were rinsed with distilled water to remove the excess stain and observed under (JEOL JEM-2100F) model TEM instrument.

The TEM micrographs which revealed the formation of agglomerated non-uniform CuO NPs with spherical, prismatic and near spherical shapes with average size of 15 nm. All these near spherical particles with varying sizes from 10.5 nm to 15.7 nm. The ring like pattern corresponding Selected Area Electron Diffraction (SAED) confirming the highly crystalline nature of the synthesized CuO NPs using PCB (Nagar and Devra, 2018).

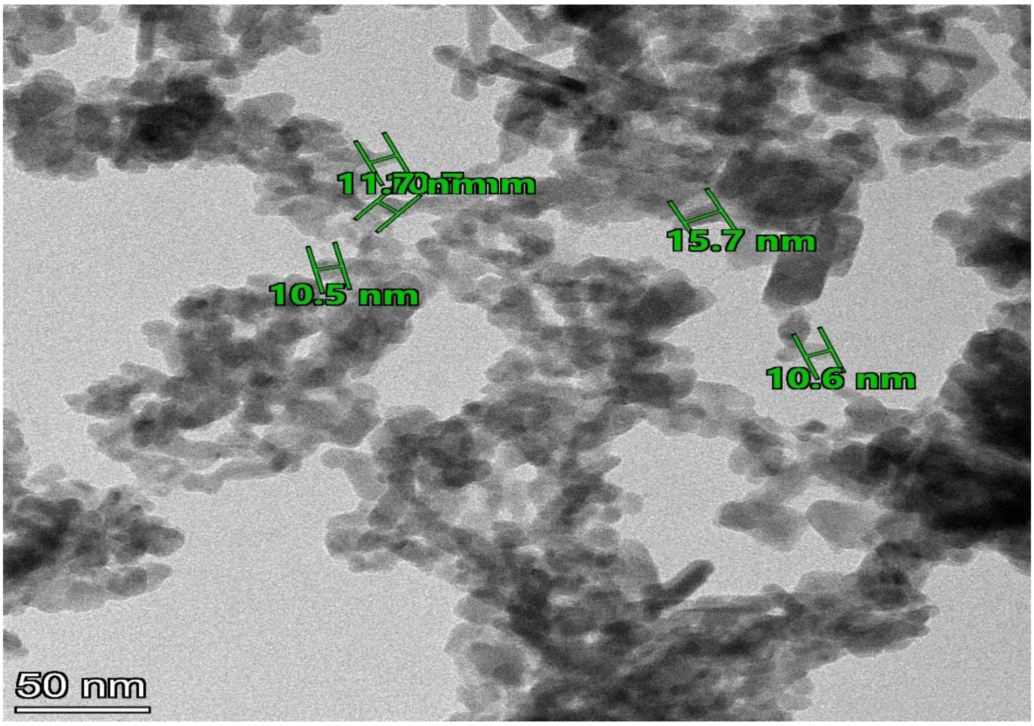


Figure 5: Transmission electron microscope (TEM) image at higher magnification (50nm) showing CuO NPs synthesized from PCB

Application of copper nanoparticles: Antioxidant activity of CuO Nanoparticles

The antioxidant activity of CuO NPs synthesized from E- Waste was tested using DPPH as a negative control and ascorbic acid as the standard. The 2,2-diphenylpicrylhydrazyl (DPPH) assay is widely used in antioxidants to evaluate the properties of compounds for scavenging free radicals. The concentrations of standard ascorbic acid, ***Parthenium hysterophorus*** Plant extract and CuO NPs were prepared with various concentrations of 25 **µg/ml**, 50 **µg/ml** , 75 **µg/ml** , 100 **µg/ml** , 125 **µg/ml** , 150 **µg/ml** , 200 **µg/ml** , 250 **µg/ml** individually. The antioxidant activity test was carried out at a wavelength of 517 nm. The results of the antioxidant activity of CuO NPs, plant extract and standard ascorbic acid can be seen in Table 2. The comparative graph of all solutions are presented in figure 6.

Antioxidant activity is expressed by the percentage of DPPH inhibition, 6 namely the ability of antioxidants to reduce DPPH free radicals. The IC_{50} value indicates the strength of a compound as an antioxidant, the smaller the IC_{50} value, the stronger the antioxidant activity. Ascorbic acid shown the lowest inhibitory concentration (IC_{50}) of 116 µg/mL, followed by 158.5 µg/ml of the green synthesized CuO NPs and 196.2 µg/mL of the plant extract. The method is based on the spectrophotometric measurement of the DPPH concentration change resulting from the reaction with an antioxidant. The DPPH radical contains an odd electron which is responsible for the absorbance at 517 nm and also for visible deep purple color. When DPPH accepts an electron donated by an antioxidant compound on the surface of Au-NPs solution, the DPPH is decolorized which can be quantitatively measured from the changes in absorbance.

S No	Concentration in µg/ml	Ascorbic acid	0.1 M CuONP	Plant extract
1	25	10.64483	7.471853	3.943603
2	50	19.34493	12.79427	5.444797
3	75	26.81679	17.7755	9.982497
4	100	37.56397	26.03207	14.55432
5	125	53.73593	35.89901	28.71331
6	150	66.93961	47.28762	44.78291
7	200	75.63971	59.80894	50.95827
8	250	88.53634	77.8915	57.4407

Table 2: Results % DPPH inhibition study for anti-oxidant activity

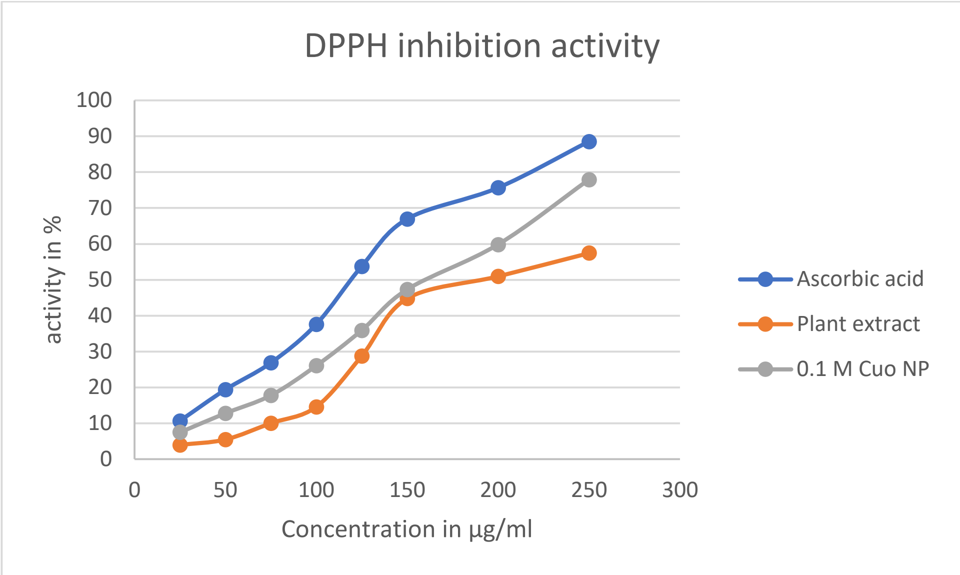


Figure 6: Comparative graph for % DPPH inhibition study for anti-oxidant activity

II. Conclusion

The antioxidant activity of copper nanoparticles was studied by DPPH radicals scavenging assay. It showed **88.5**% of scavenging activity which indicates synthesized copper nanoparticles is a potential anti-oxidant agent. The UV spectroscopy results indicate that maximum absorption between 260 and 290nm which indicate the formation of copper oxide and TEM analysis indicated the formation of non-agglomerated copper nanoparticles.

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