GREEN SYNTHESIS OF CUPROUS OXIDE NANOPARTICLES

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ABSTRACT

Cuprous oxide Nanoparticles (Cu_2O NPs) were syntheised by using benign solvents such as plant leaf extracts of Camellia sinensis (Green tea). The structure and morphology of the nano particles were studied using X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Ultraviolet-visible spectroscopy (UV-vis) and Thermogravimetric analysis (TGA). Antibacterial activity of the nanoparticles was also studied.

Keywords: ***Cuprous Oxide Nanoparticles (Cu_2ONPs), Fehlings Solution, Green Synthesis, Camellia Sinensis***

I INTRODUCTION

In the recent years, much attention has been paid to metal oxide nanoparticles that give rise to unique electronic and optical properties that are useful for a variety of new technologies in optoelectronic devices, chemical sensors [1, 2], molecular catalysts [3] and magnetic materials. Metal oxide nanoparticles (NPs) have shown great potential in the field of sensing, optoelectronics, catalysis, solar cells [4] and so on, due to their physical and chemical properties different from those in the bulk material. Among all the metal oxides, copper oxide an interesting p-type semiconductor [4] has attracted more attention due to its unique properties. In this work we have focused on developing an eco-friendly 'green synthesis' route [5-8], for the production of (Cu_2O NPs). We have followed the formation of copper (I) oxide from the basis of the Fehling's test and Benedict's test for reducing sugars. Hence in the proposed method Camellia sinensis plant leaf extracts were used as stabilizing agents which act as biotemplates for the synthesis of Cu_2O NPs. The structural and anti-bacterial properties of the Cu_2O NPs have been evaluated.

II MATERIALS AND METHODS

Copper (II) sulfate pentahydrate (Central Drug House (P) Ltd.,), Potassium sodium tartarate tetrahydrate A.R. (Nice Chemicals (P) Ltd.,), Sodium hydroxide Pellets (Avra Synthesis (P) Ltd.,) distilled water were used in the nanoparticle synthesis with the extract. The Camellia sinensis Tetley brand was procured from Nilgiris Super Market. Stock culture of bacteria for antibacterial studies was obtained from (Science House, Chennai). Mueller-Hinton agar, Nutrient broth, disposable sterile petridishes, cotton swabs, sterile saline, test tubes was purchased from HiMedia Laboratories Pvt Ltd.

2.1 Preparation of Fehling's Solution

Fehling's solution which is comprised of equal parts of Fehling's A and B were prepared as follows:
*Fehling's A***:** *P*repared by dissolving of Copper (II) sulfate pentahydrate (14 g) in distilled water (200 mL). *Fehling's B***:** Prepared by dissolving of Potassium sodium tartarate tetrahydrate (70 g) and sodium hydroxide (30 g) in distilled water (200 mL).

2.2 Preparation of Leaf Extracts

Camellia sinensis leaves (10 g) were thoroughly washed and then boiled in 50 ml of deionised water for half an hour. The resulting extract was cooled and used as the extract solution.

2.3 Synthesis of Cu_2O Nanoparticles

Ten mL of the green tea leaf extract was added to 10 mL of Fehling's solution. After 10 minutes, the color of the solution changed from blue to brick red, indicating the formation of cuprous oxide nanoparticles. The product was washed thoroughly with distilled water and then calcined at 52-60^o C.

2.4 Antibacterial activity

The antibacterial activity of the CuGT nanoparticles was studied by the broth inoculation method. Initially a broth is prepared by autoclaving 2.6 g of nutrient broth in 100 ml in a conical flask. The broth is divided between three test tubes –the standard, the control and the test. One ml of a 20 mmol suspension of the nanoparticle (CuGT) was added to the standard and the test and one loopful of bacteria were then added to the control and the test .The tubes were then incubated in an incubator for 24 hours. The growth and inhibition of bacteria was monitored by taking absorbance readings during a period of 18 to 22 hours using a colorimeter.

III RESULTS AND DISCUSSION

The addition of Fehling's solution to the leaf extract containing carbohydrates as a major component, which has aldehyde groups, may cause the reduction of copper ions which results in the formation of brick red Cu_2O precipitate. The chemical reaction which occurs can be seen in the following equation

$$RCHO + 2Cu^{2+} \rightarrow 2H_2O + RCOOH + Cu_2O + 4H^+$$

The UV-Vis spectrum of the synthesized CuGT nanoparticles was carried out on a JASCO UV- Vis spectrophotometer is shown in Fig. 3.1. Confirmation of the synthesised CuGT nanoparticles was exhibited by the absorption band at 466 nm which is consistent with nanoparticle absorption behavior.

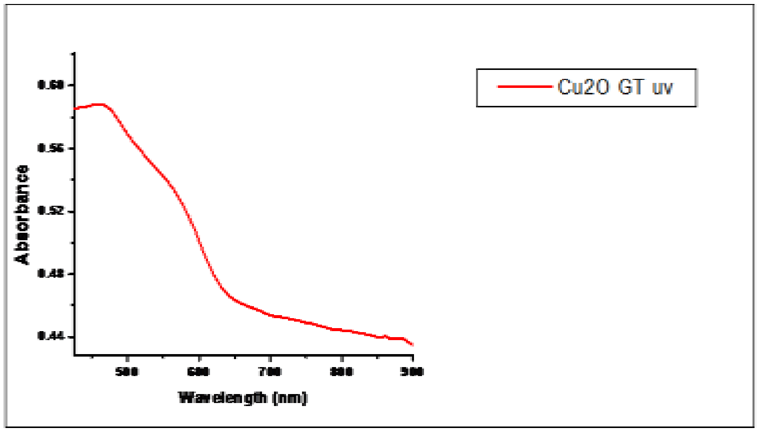


Fig.3.1 UV absorption spectrum of CuGT

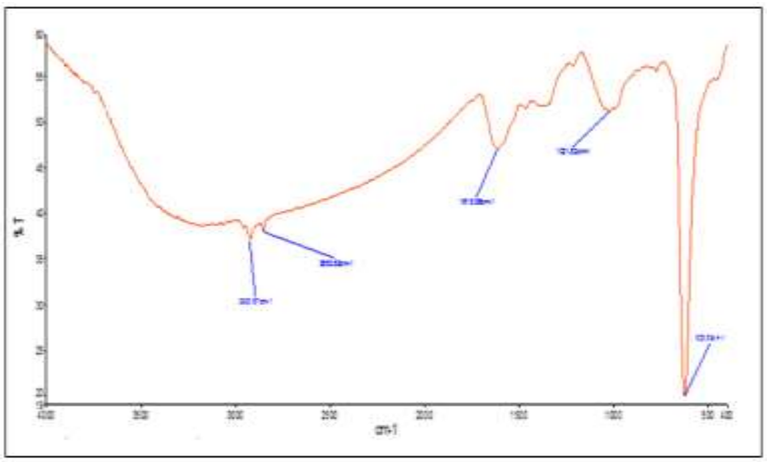


Fig 3.2 IR spectrum of CuGT

The IR spectrum (Fig.3.2) was taken using a Perkin Elmer FT –IR instrument operating at a resolution of 4000-400 cm^{-1} in the percent transmittance mode. In addition to the absorption bands of the biomolecules used as reduction and stabilization(capping) agents, the absorption peak at $622cm^{-1}$ indicate the formation Cuprous oxide nanoparticles (CuGT).

XRD spectrum of the as prepared CuGT nanoparticles was carried out using XRD (Bruker AXS D8 Advance) for 2θ values ranging from 10 to 80° using CuKα radiation. Good crystalline peaks were observed. In CuGT, the 2θ values at 29.50, 36.38, 42.29, 52.47, 61.43, 73.62 and 77.50 were observed. The spectrum (Fig. 3.3) confirmed the cubic cuprous oxide structure of CuGT with the lattice constants of a = 4.274 Å, which are in good agreement with the JCPDS No. 65-3288(a = 4.260 Å). The average particle size (D) of synthesised nanoparticles was calculated using the well known Scherrer formula. The calculated value of D was 34.36 nm.

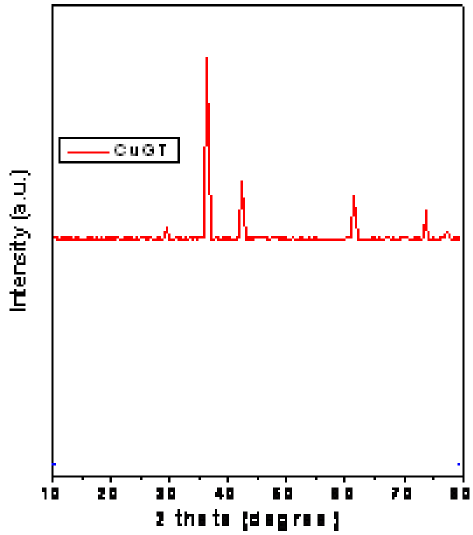
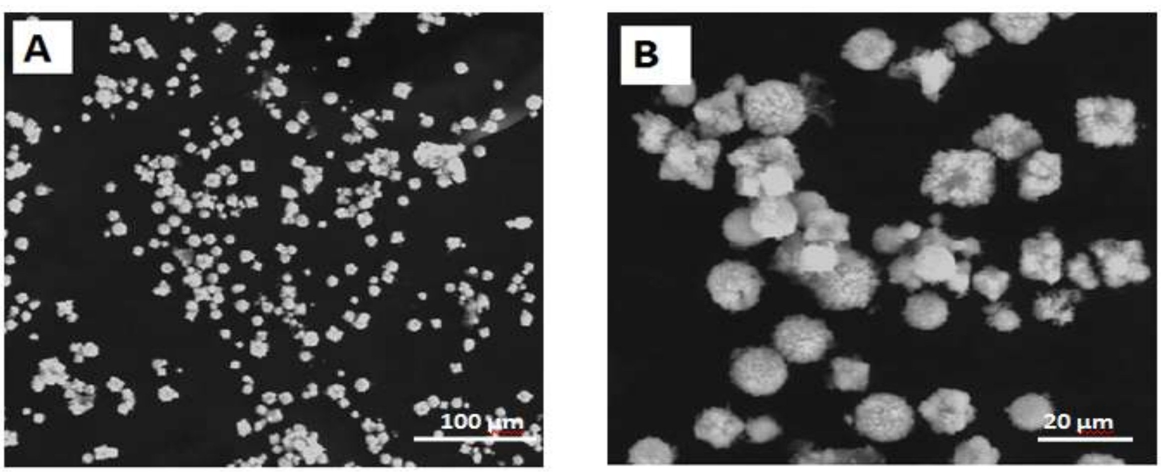


Fig 3.3. XRD pattern of CuGT

The physicochemical properties of Camellia sinensis acts as a biotemplate. The SEM image by green synthesis method explains the external morphology of the Cu_2ONPs

(CuGT). The SEM images of CuGT studied using SEM (Quanta 200 FEG scanning electron microscope) (Fig 3.5) indicate very little agglomeration with a mixture of truncated cubes and spheres.



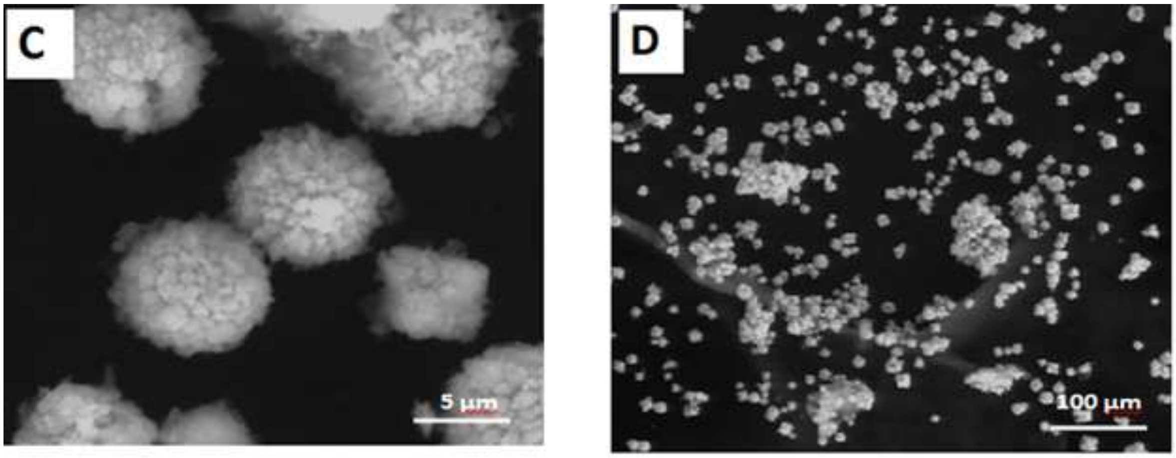


Fig 3.4 SEM images of CuGT

The thermogravimetric analysis of CuGT was performed on a Perkin–Elmer Thermo gravimetric analyser. The TGA (Fig. 3.5) showed a 30 % loss in weight starting around 100°C indicating the loss of water and other volatile components in the Camellia sinensis extract which was used as a stabilizing agent

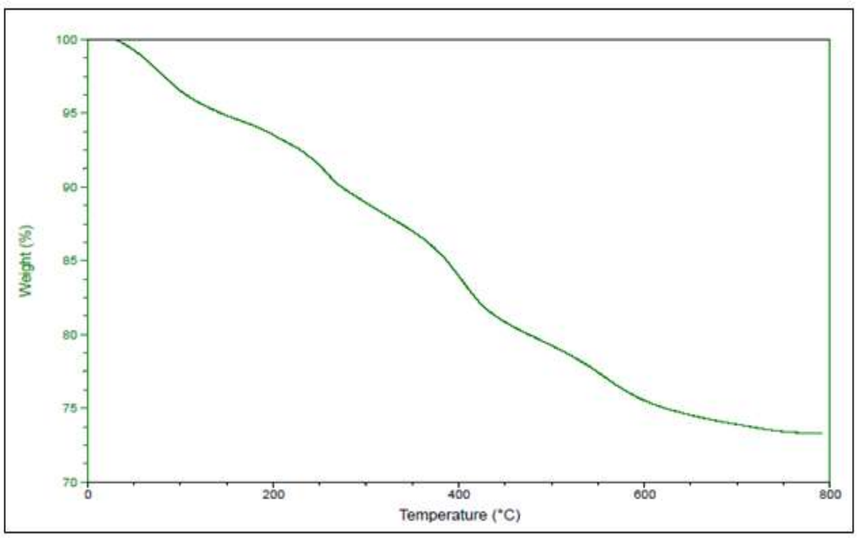


Fig 3. 5 Thermogravimetric curve of CuGT

The presence of any antibacterial activity on the synthesized Cu_2O NPs was first detected in the surface inoculation method (Fig.3.6)

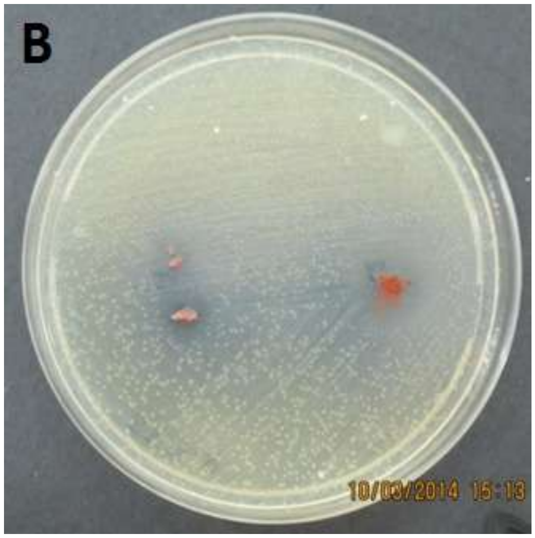
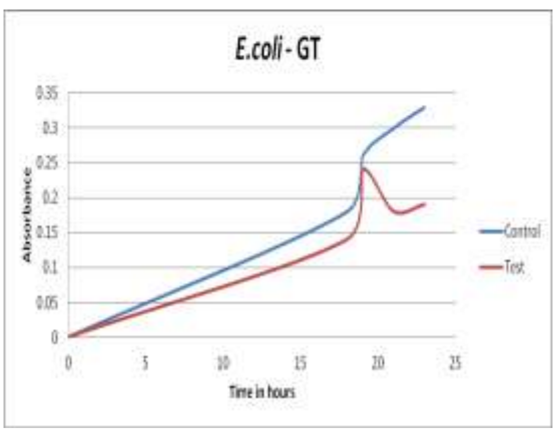


Fig. 3.6 Antibacterial activity of CuGT on (a) *E.coli* and (b) *S.aureus*

In order to obtain more accurate information of the antibacterial property of CuGT via a time-dependent study , the broth inoculation method was carried out using five bacteria : *E.coli, S.aureus* ,*K.pneumoniae* ,*P.mirabilis* and *B.cereus* .This method was used to determine the antibacterial properties of the synthesized nanoparticles by comparing the growth of the bacteria in the control tube (containing the broth and bacteria) and that in the test(containing the broth, bacteria and nanoparticle suspension). The growth of the same was studied colorimetrically for a period of 18 -22 hours.



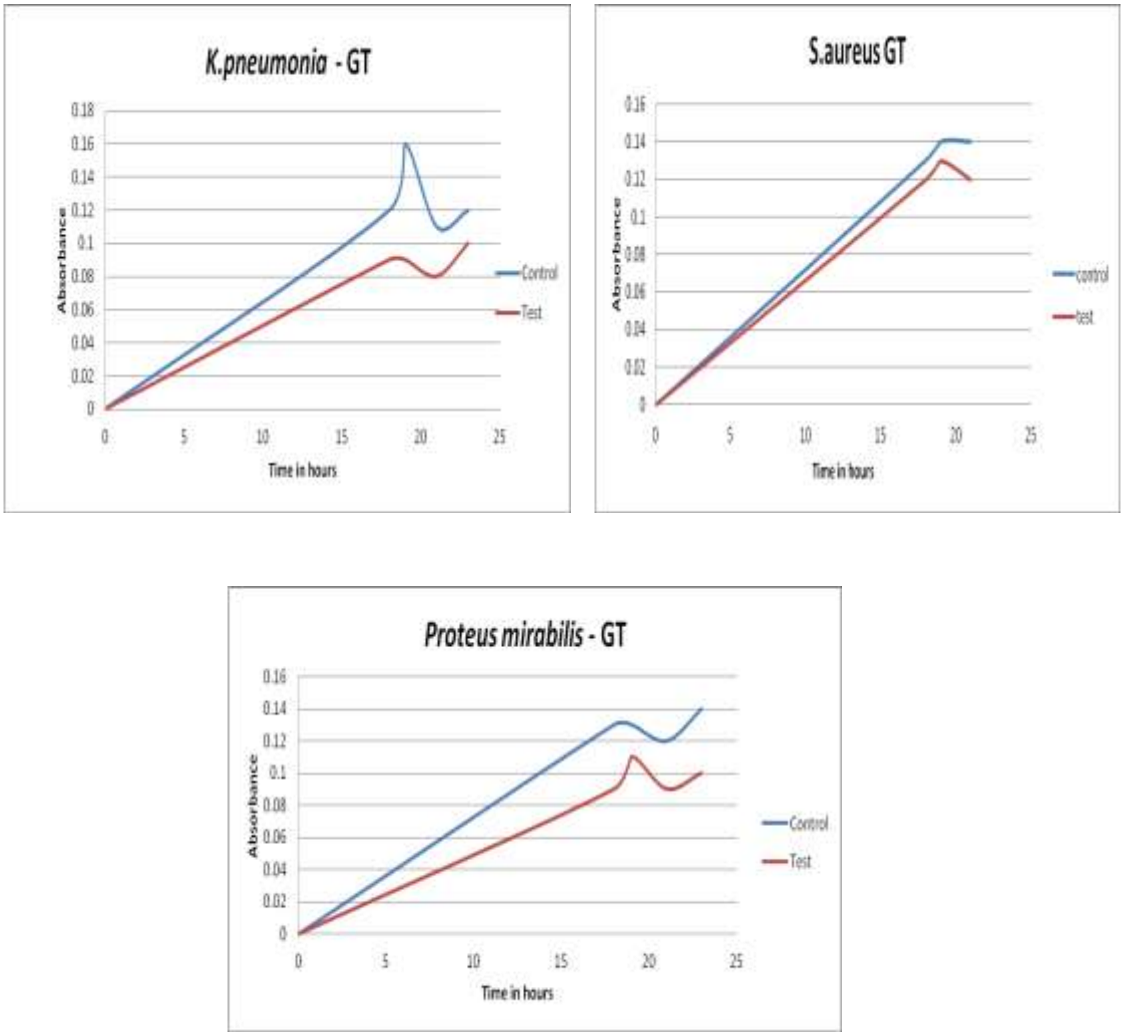


Fig.3.7Antibacterial studies of CuGT

The antibacterial studies of CuGT(Fig.3.7) showed sensitivity to both Gram positive and Gram negative bacteria. CuGT nanoparticles exhibited antibacterial action towards *E.coli* (gram negative) and *B.cereus* (gram positive).

IV CONCLUSION

The green chemistry approach used in the present work for the synthesis of Cu_2O nanoparticles is simple, cost effective and the resultant nanoparticles are highly stable and reproducible. Their antibacterial activity represents a significant advancement in the nanomaterials field, with realistic applications.

REFERENCES

[1] M. Kooti and L. Matouri, Fabrication of Nanosized Cuprous Oxide Using Fehling's Solution, *Transaction F: Nanotechnology* 17*(*1), 2010, *73-78*.

[2] Yakui Bai, T.Y., Qing Gu, Guoan Cheng, Ruiting Zheng Shape control mechanism of cuprous oxide nanoparticles in aqueous colloidal solutions. 2012, 35-42

[3] Neha Topnani .; Wet Synthesis of Copper Oxide Nanopowder. *International Journal of Green Nanotechnology*: Materials Science & Engineering1(2,) 2009.), M67-M73.

[4] Rajesh Ramanathan.; Bhargava, Suresh K.; Vipul Bansal.; Biological Synthesis of Copper/Copper Oxide Nanoparticles,*Chemca*., 2011, 18-21

[5] D. Gnanasangeetha, S rala Thambavani D., One Pot Synthesis of Zinc Oxide Nanoparticles via Chemical and Green Method. *Research Journal of Material Sciences* 1(7) 10 ,2013: p. 1-8.

[6] Chandra.S.; A.Kumar,.;P.K. Tomar, Synthesis and characterization of copper nanoparticles by reducing agent. *Journal of Saudi Chemical Society*, 2014. 18(2): p. 149-153.

[7] Rajesh Ramanathan.; Bhargava, Suresh K.; Vipul Bansal.; Biological Synthesis of Copper/Copper Oxide Nanoparticles. *Chemca*. 2013, 18-21

[8] K.S Kavitha.; D Rakshith., H.U Kavitha.; H.C Yashwantha Rao.; B.P Harini.; Satish S, Plants as Green Source towards Synthesis of Nanoparticles.International *Research Journal of Biological Sciences*, 2013