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GREEN SYNTHESIS OF COPPER NANOPARTICLES USING MENTHA SPICATA LEAVES EXTRACT

Prasad S. Kshirsagar* and Darshana S. Mote

SCES's Indira College of Pharmacy, Tathawade, 411033, Pune, India.

***Corresponding Author: Prasad S. Kshirsagar**

SCES's Indira College of Pharmacy, Tathawade, 411033, Pune, India.

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ABSTRACT

Objectives: The green synthesis of nanoparticles having eco-friendly approaches and generating interest & scope for researchers so present study focusses on the green synthesis of stable copper nanoparticles. **Methods:** This method involved preparations of leaf extract of Mentha Spicata in double-distilled water which act as a bioreducing agent. This synthesis involved addition of reducing agent to Cuso4 solution at different maintained temperature by using hot plate with magnetic stirrer. **Result:** During addition visual color change is observed from light sky blue to dark reddish-brown indicates the formation of the Copper nanoparticle. Prepared Nanoparticles were characterized using U.V visible spectrometry, Fourier transforms infrared spectroscopy (FTIR), X-Ray diffraction (XRD) and Transmission Electron Microscopy (TEM). **Conclusion:** Mentha Spicata leaf extract reduce the copper ions into copper nanoparticles within 25-30 minutes of time interval. By using this green synthesis there is a rapid, stable, eco-friendly synthesis of copper nanoparticles achieved.

KEYWORDS: Copper Nanoparticles, Metallic nanoparticles, Copper Sulphate, Green Synthesis, Mentha Spicata, Mentha, Bio reduction.

INTRODUCTION

Nanoparticles are fundamental building blocks of nanotechnology and wide range of research applications. Nanoparticles exhibit enhanced properties based on their specific characteristics such as size and morphology.[1,2] Research in Nanoparticles has considerable scope because of unique properties & application in different areas like biotechnology, physics, chemistry, material science, and engineering medicine.[3] Nanoparticles are synthesized by physical and chemical methods; these are suffering from drawbacks such as hazardous reaction condition, longer time, expensive reagent, tedious process to isolate NPs.[4,5] So there is scope to develop new methods for the synthesis of NPs which required less drastic reaction condition, inexpensive reagent, and eco-friendly. Among the nanoparticles copper nanoparticle gained much importance because of less cost of preparation, and has excellent physical and chemical properties.[6] In literatures, the copper nanoparticles were synthesized from various methods like vapor deposition,[7] electrochemical reduction,[8] radiolysis reduction,[9] thermal decomposition,[10] chemical reduction of Copper metal salt,[11] and room temperature synthesis using starch and hydrazine hydrate.[12] In recent, green synthesis of CuNPs was achieved using microorganisms,[13] plant extract.[14] In various literature cu nanoparticles are synthesized using various plant leaves extract like (a) *Ocimum Sanctum*[3] (b) *Madhunashini*[15] (c) Aegle *Marmelos*[3] (d) Tea[16] (e) *Arevalanta*[17] *(f) Eclipta prostrate.*[18]

Mentha also called as Mint from Greek Mintha, is a genus of family Lamiaceae. It is estimated that 13-15 species exist, and the exact distinction between species is still not clear. Hybridization between some of the species are natural, many other hybrids as well as numerous cultivars, are known.[19] Leaves are crumpled, opposite, ovate - lanceolate, 3-7 cm long. The leaves are sessile with bright green colour free from purple as shown in Figure1.

Inflorescence is slender, interrupted cylindrical spikes are crowded, lanceolate spikes with 7-10 cm long bracts. M. Spicata contains volatile oils, resins, tannins, coumarins, flavonoids, steroids, alkaloids.[20] The clinical effectiveness of many existing antibiotics is being threatened by rapid emergence of multidrug resistant pathogens[21] down the ages there has been an increasing interest in the use of plant extracts and essential oils as alternative remedies for the treatment of various infectious diseases.

MATERIALS AND METHODS

Chemicals

Copper sulphate, alcohol, tween, distilled water were obtained from department of pharmaceutical chemistry SCES's Indira college of pharmacy, Pune 33.

Collection of the plant sample

The Mentha Spicata leaves were collected from farm in and around Khed - Shivapur, Pune Maharashtra, India The plants were identified and studied according to their families. Fresh plant materials were collected and washed under tap water, shade dried and then homogenized to fine powder and stored in airtight bottle.

Preparation of Mentha Spicata leaves extract – Herbal Extract

The green fresh leaves of Mentha Spicata were collected thoroughly washed with normal water and then with double distilled water to remove impurities. The washed leaves were dried to remove the surface water, then accurately weigh about 5 g of leaves and keep at room temperature then the prepared solution was initially filtered through normal filter paper thereby after using whatmann's filter paper to get clear solution as shown in figure 2. This filtrate then freshly used for further work.

Preparation of 0.01 M CuSo4 solution

Weigh accurately about 1.25 g of $CuSO_4$ and dissolved in 500 ml of distilled water to get 0.01M $CuSO_4$ solution shown in figure 3.

Green Biosynthesis of cu nanoparticles using leaves of mentha spicata

10 ml herbal extract of Mentha Spicata leaves was added by using micro pipette into 400 ml of 0.01M $CuSO_4$ solution under continuous stirring at temperature 60-70 ^{0}C for 30 minutes. There is change in sky blue color of $CuSo_4$ solution to greenish yellow colour then to brown colour which indicates formation of copper nanoparticles as shown in figure 4. Then placed the solution in incubator for 24 hours then centrifuged it at 4500 RPM for 30 minutes then pellets are taken out and dispersed in deionized water & then dried in oven for further analysis and characterization purpose. Then Tween 80 was added for preservation purpose that maintains dispersibility and preventing aggregation of copper nanoparticles.

Characterization of Cu nanoparticles

Copper nanoparticles were characterized by UV spectroscopy using Jasco V-730 double beam UV-Visible Spectrophotometer, FTIR spectroscopy using Jasco Infra-red spectrophotometer by KBr press pellets method.The crystal structure and size of nanoparticles are analysed by XRD by X Ray Diffractometer normal mode at Physics Department Savitribai Phule Pune University GaneshKhind Pune 411007.TEM analysis carried on model TEM CM-200 (Philips) 2.4 Aoat IIT Bombay IIT Bombay Sophisticated Analyatical Instrument Facility (SAIF).

RESULT AND DISCUSSION

UV visible spectroscopy

Nanoparticles show specific optical properties which response to the morphology like shape and size, concentration, agglomeration state and refractive index near nanoparticles surface so UV Visible spectroscopy is important for identifying characterizing and studying nanoparticles.[22]

In UV Spectroscopy small amount of synthesized nanoparticles taken out and sonicated then UV analysis carried out by using blank as mixture of extract and $CuSO_4$ solution to minimise solvent effect. Surface Plasmon vibration of copper nanoparticle gives wavelength of maximum absorption λ_{max} at 560nm as shown in figure 5.

Fourier transform infrared spectroscopy

The FTIR spectrum as shown in figure 6 suggest that copper nanoparticle surrounded by biomolecules likes protein amino acid and metabolites of terpenoids and functional groups of alcohol, ketone, aldehyde, amine and carboxylic acid.

The FTIR studies confirmes the presence of C=C aromatic double bond and OH group which give rise to phenolic group. Phenolic groups have the ability to bind metal that indicated phenol was from copper nanoparticles, which is due to capping of nanoparticles; this provides stability and prevent agglomeration.[3,23]

Sr. No	FTIR peak cm^{-1}	Functional groups
1)	3343	N-H stretching
2)	2970	Carboxylic O-H
3)	2877	Other C-H bond
4)	2362	Triple bond region C≡C
5)	1748	Carbonyl group C=O
6)	1412	Double bond aromatic C=C

XRD Analyasis

The crystal structure and size of the NPs are verified by XRD analysis

XRD analysis carried out using X Ray Diffractometer normal mode at Physics Department Savitribai Phule Pune University GaneshKhind Pune 411007.

XRD pattern of cu nanoparticles synthesized from mentha spicata was shown in figure 7. Its clearly indicates crystalline nature of nanoparticles. The diffraction angle observed at 45.78^0

The size of nanoparticles estimated which is found to be 22 nm using Debye Scherrer equation which indicates high surface area to volume ratio of copper nanoparticles.[3]

$$D=k\,\lambda/\beta\,COS\theta$$

Where

k =Scherer's constant (shape factor) which is 0.9 to 1

λ= wavelength used of xrays radiation

β= width of xrd peak at half height

θ=Bragg angle of diffraction

TEM analysis

TEM analysis carried out on model TEM CM-200 (Philips) 2.4 A^{o}at IIT Bombay Sophisticated Analyatical Instrument Facility (SAIF)

TEM images of copper nanoparticle which is prepared by mentha spicata leaves extract was shown in figure 8. The images indicates formation of sperical shape crystalline copper nanoparticles. Previously synthezied spherical copper nanoparticles were reported.[24,25]

Fig. 1: Mentha spicata leaves.

Fig. 2: Herbal extract of menthe.

Fig. 3: Copper sulphate solution.

Fig.4: Synthesized CuNP solution.

Different shades of copper nanoparticles during synthesis

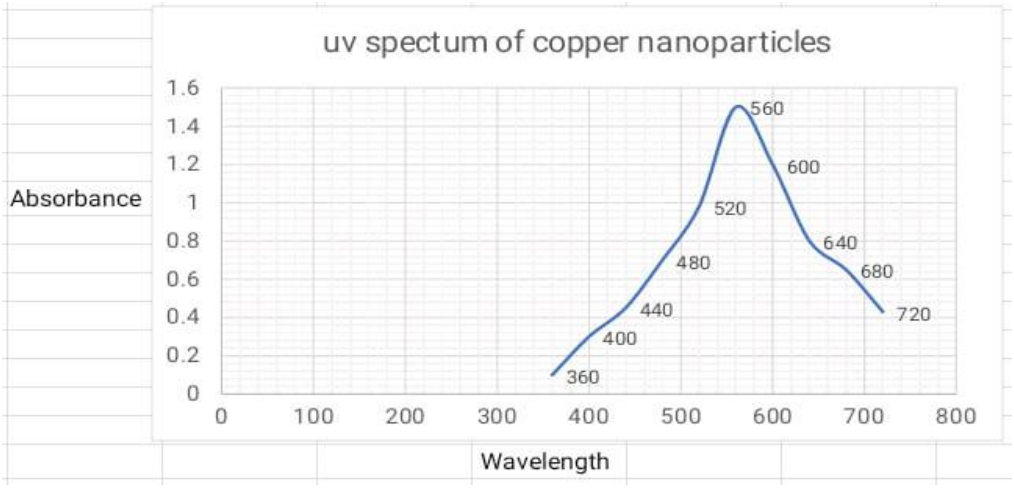


Fig. 5: UV Visible spectrum of copper nanoparticles.

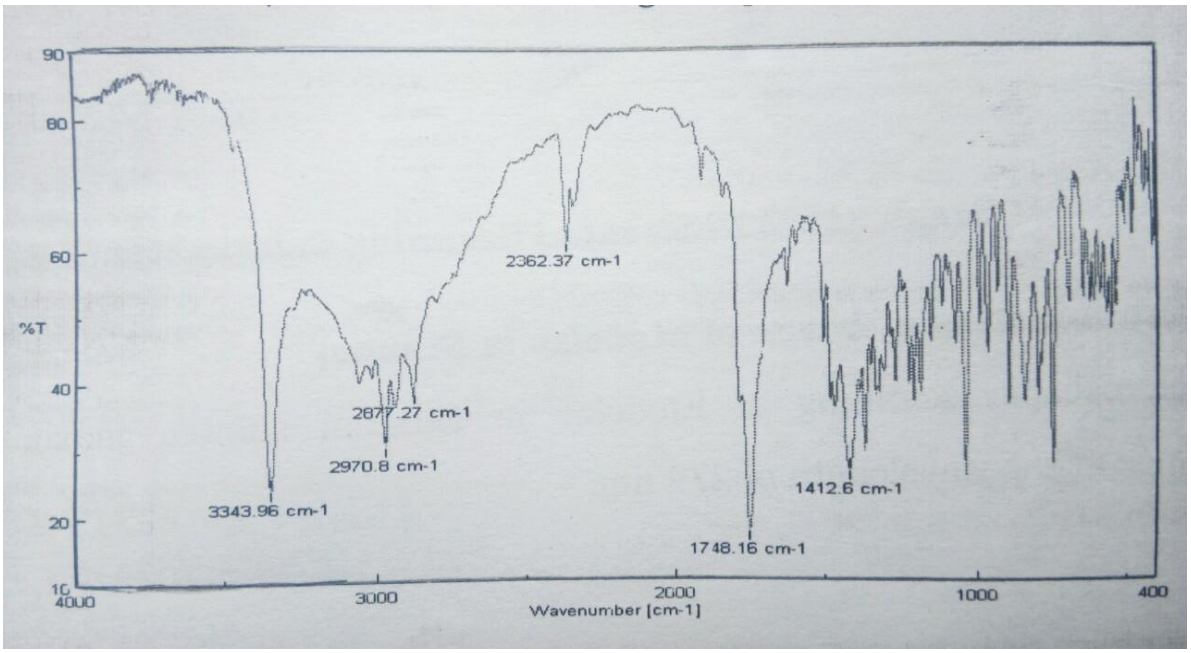


Fig. 6: Fourier transform infrared spectrum of copper nanoparticles.

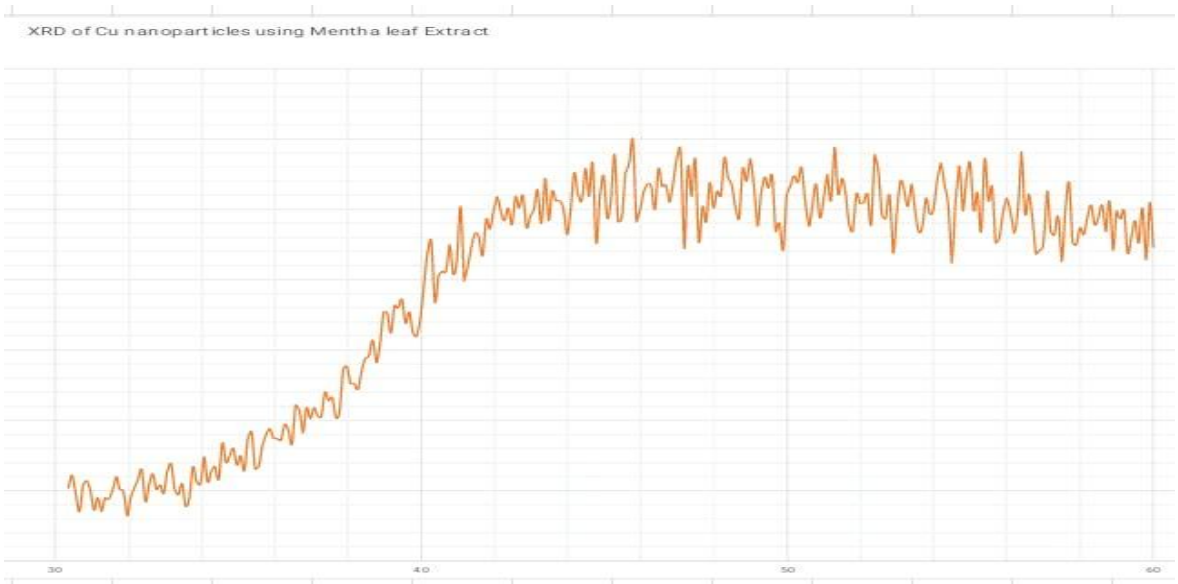
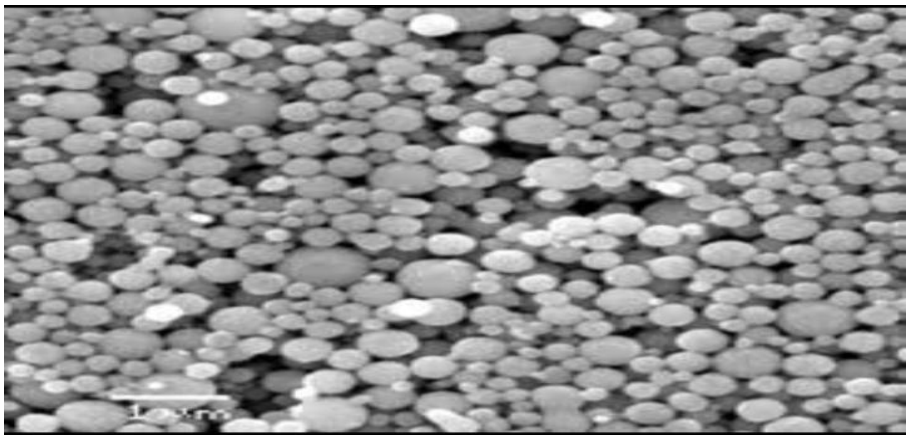
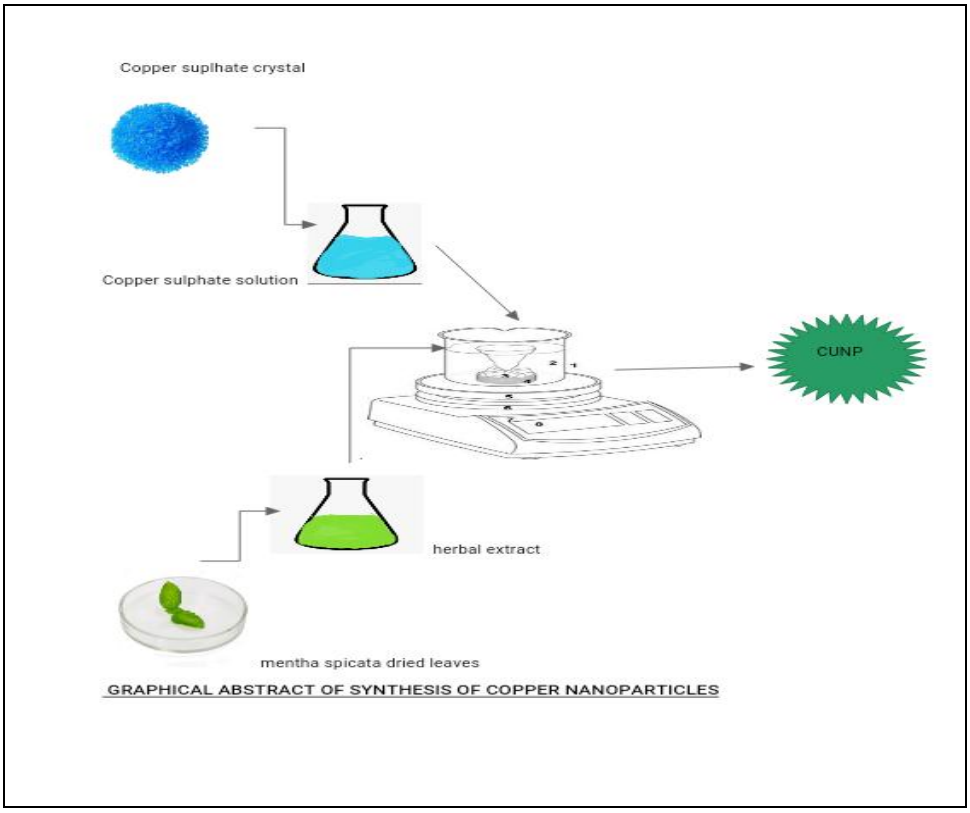


Fig. 7: XRD interpretation of copper nanoparticles.

TEM: images



Pictorial Abstract



SUMMERY
Our present work focused on reduction of copper sulphate to copper nanoparticles by using mentha spicata leaves extract by bio reduction method. Synthesized copper nanoparticles were characterized by various methods like UV, FTIR, XRD and TEM.

CONCLUSION
In green synthesis of copper nanoparticles from leaves extract of Mentha spicata. Formation of nanoparticle was identified initially by colour change of copper sulphate solution light blue to dark reddish brown then the nanoparticles characterized by UV spectroscopy, IR spectroscopy, X-ray Diffraction and TEM analysis. The green source used for synthesis acts as reducing and stabalising agent. It is safer and environmental friendly method than other physical or chemical methods as no toxic reagent used during synthesis. Also the procedure used is easy to perform in laboratory at room temperature and pressurs. Starting material for synthesis is easily available so this method having ecofriendly approaches.

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CONFLICT OF INTEREST
We authors declare no conflict of interest.

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