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Green Synthesis of Copper Nanoparticles and its Characterization

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Abstract: **The green nanotechnology is generating interest in researchers for the synthesis of nanoparticle in a simple, cost effective, less toxic and ecofriendly manner. The present study reports the biosynthesis of copper nanoparticle using the leaf extract of *Ocimum sanctum*. The color change in the *Ocimum* leaf extract when copper sulphate solution is added indicates the presence of copper nanoparticle. The effect of temperature and time of incubation on the biosynthesis of Cu NP were noted. The characterization of the biosynthesized copper nanoparticle was done by UV Vis spectrophotometer, Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD).**

Index Terms: **Copper nanoparticles, Cu NP, Nanoparticle, X-ray Diffraction, XRD, FTIR, UV-Vis spectroscopy**

I. Introduction

Nanoparticles (NPs) are ultra-small units in the size range of 1 to 100 nanometers. Because they are very small, they are highly mobile and also chemically active because of the increased surface area due to its small size. It is also known that the molecules and atoms in metals or non-metals in nano range show drastic change from the parent bulk material. Due to these characters they have so many practical applications in various fields such as medicine, industry, environmental remediation etc. They have the ability to limit potentially toxic metal concentrations. The interaction of nanoparticles with various materials is very much different from the corresponding metal of greater size range. The production of nanoparticles is increasing steadily because of the prospectus applications. Last decade has seen a substantial increase in the nanoparticles production of about 25 fold between 2005 and 2010 (PEN 2013).

But chemical synthesis of nanoparticles is reported to have several problems. Studies have shown that preparation of some nanoparticles need thermal processing. And this processing may alter the physicochemical properties of the nanoparticles synthesized including their density, crystal structure and the presence of surface contaminants, (Karakoti et al., 2013). The chemically synthesized nanoparticles, also called engineered nanomaterials (ENM) in case if used for biological applications, there is a chance of generation of toxicity. Release of these particles to the environment should also be done with caution. Karakoti et al., (2013) have noticed that very small changes in room temperature during synthesis as well as during storage may lead to unpredictable as well as non-reproducible properties in nanoparticles.

Ecofriendly technologies for production of nanoparticles were given special attention due to the aforementioned facts. The objectives were to produce environmentally benign and nontoxic nanoparticles using green synthesis and biotechnological tools, (Joerger et al., 2000; Chauhan et al., 2012). The advantage of green synthesis of nanoparticles over their chemical synthesis are due to the fact that the nanoparticles prepared are with diverse nature, greater stability and appropriate dimensions and they are synthesized using a one-step procedure. The undesired conditions during chemical synthesis are eliminated and conditions like physiological temperatures, pH, pressure are only used in green synthesis with biological components acting as reducing and capping agents and that too at a negligible cost (Ingale and Chaudhari, 2013; Noorjahan et al., 2015).

Copper nanoparticles find application in industries, medicine, electronics etc. due to the peculiar properties like high electrical conductivity, low electrochemical migration behavior high melting point and after all its low cost. There are reports (Tanilvanan et al., 2014) to show that copper nanoparticles can be used as alternatives for noble metals such as in inkjet printing, for organic transformations, gas phase catalysis, photocatalysis and electrocatalysis, (Gawande et al., 2016). Since Cu-nanoparticles have biomedical applications, the solvent, stabilizing agent as well as reducing agent used for its synthesis should be non-toxic. This signifies the biosynthesis of Cu-nanoparticles which is a bottom-up method of nanoparticle

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synthesis.

Realizing the significant applications of Copper nanoparticles in the above mentioned fields and also the problems associated with the chemical synthesis of the said nanoparticles, this study was undertaken with the objective to biosynthesize Copper nanoparticle using the extract of *Oscimum sanctum* (which is used as a source of bioreduction and stabilizers) and to characterize the particles synthesized using techniques like UV-VIS spectrophotometer, Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD).

I. EXPERIMENTAL

A. Preparation of Leaf Extract

For the synthesis of copper nanoparticles *Ocimum sanctum* leaves were used. About 50 g of leaf was collected, washed thoroughly in distilled water, cut into small pieces and added about 50 mL of double distilled water into it. It was heated in water bath for about 10-15 minutes at 80ºC. The resultant extract was filtered using Whatman filter paper no1 and stored in refrigerator for further use.

B. Synthesis of copper nanoparticles

For nanoparticle synthesis, about 25mL of *Ocimum sanctum* leaf extract was added to 100 mL of 1mM copper sulphate solution in a 250 mL conical flask. The solution was incubated for a period of 10 hours. Three replica were placed each at three different temperatures (27°C, 40°C and 80°C). The solution thus obtained was centrifuged at 12000 RPM for 15 minutes. The resultant pellet was washed with distilled water and the copper nanoparticles are dried at hot air oven at 80ºC. Concentration of Cu nanoparticles obtained at three different temperatures and at different time intervals were also determined.

C. Characterization of Nanoparticles

Characterization techniques help us to understand the specific properties of the nanocrystals studied in an accurate manner which is reliable to understand the measured values. The synthesized nanoparticles were subjected to various characterization studies to understand the specific properties such as optical, structural, morphological, elemental composition, particle size and functional groups studies. The characterization of green synthesized nanoparticle was done by using UV-VIS spectrophotometer, Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD).

II. RESULT AND DISCUSSION

A. Biosynthesis of Cu nanoparticles

The color of the leaf extracts changes from light brown color to dark green after adding copper sulphate solution (Plate 1.). The color change indicates the formation of copper nanoparticles. The *Ocimum* leaf extract can reduce the copper ions into the copper nanoparticles within 12 hours. From the figure 1, we can see that as time increases, the concentration of Cu NP increases and reaches a plateau at 600 minutes. From Fi1 and Fig.2 we can also see that as the time of incubation increases, the quantity of nanoparticles synthesized also increases and reaches a plateau. In the current study, the effect of temperature on Cu NP synthesis was also noted and seen that the synthesis is temperature dependent and maximum synthesis occurs at 80°C.

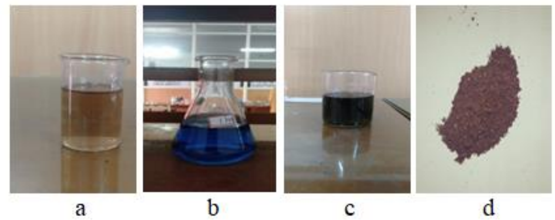

Plate 1 Visual observation of a) leaf extracts b) copper sulphate solution c) Copper nanoparticle solution d) Copper nanoparticle

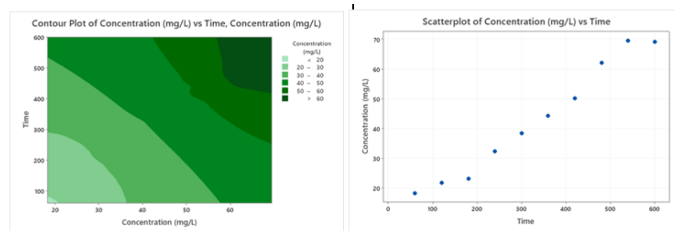

Fig. 1& 2: Contour plot and matrix plot showing concentration of Cu NP synthesized in relation to time

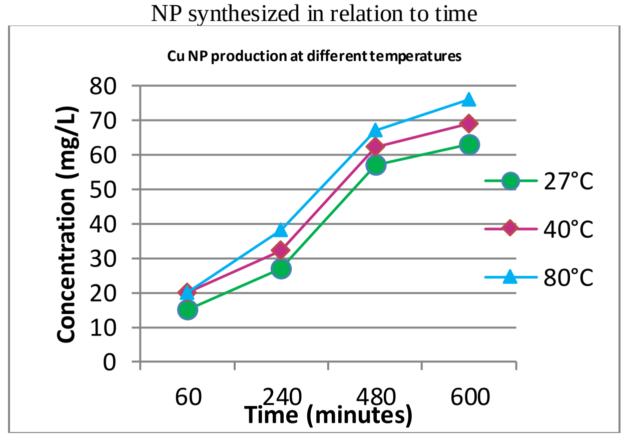

Fig. 3: Effect of temperature on Cu NP synthesis

B. Characterization of Nanoparticles

1) UV – Vis Spectrophotometer

The synthesis of copper nanoparticle was first confirmed by UV-Vis spectrophotometer (Fig 4). UV- visible spectral analyses of copper nanoparticles was done to characterize the Cu NP formed at a range of 350nm to 700nm.The maximum absorption can be seen at a range between 550nm and 600nm at about 560nm. The broadening of peak indicates that the particles are poly-dispersed. Nanoparticles of metals have free electrons (Ananthalekshmi et al., 2016). The mutual vibration of these electrons gives rise to a surface plasmon resonance (SPR) and in turn an absorption band. The figure shows peak characteristic of SPR of Cu NPs. It was also found that the peak value gradually decrease with the increase in the particle size. This is because of the fact that as the particle size increases, the particles becomes opaque (Goh et al., 2014). It is found that the spectrophotometric analysis is sensitive to the detection of larger particles or aggregates of small particles (Nakamur et al., 2013).

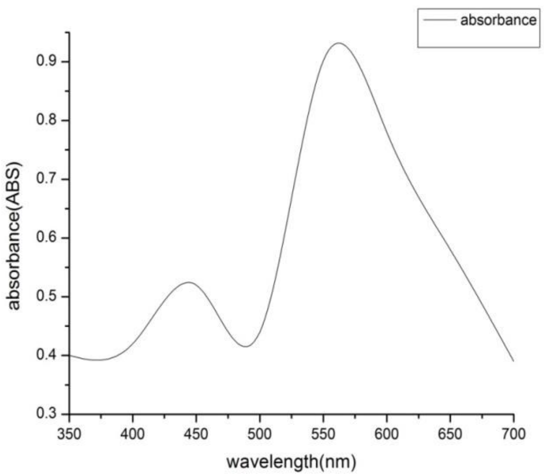

Fig 4: UV-visible absorption spectroscopy analyses of copper Nanoparticles.

2) FTIR Spectroscopy

FTIR analysis is done to determine to find the possible biomolecules in the *Ocimum sanctum* extract and the functional group or the organic compounds which are responsible for the synthesis of copper nanoparticles. The FTIR analysis is done at a range between the 4000 to 500 nm. The peak can be seen at 3799 cm^{-1}, 3141cm^{-1}, 1581cm^{-1},1405cm^{-1}, 1245cm^{-1}, 1044cm^{-1}, 854cm^{-1}, 805 cm^{-1}. The peak at 3799 cm^{-1} indicates OH group, 3141cm^{-1} indicates the carboxylic acid group, 1581cm^{-1} indicates amine group, 1405cm^{-1} shows the presence of alkanes, 1245cm^{-1} indicates alkyl ketone group, 1044cm^{-1} indicates C-O group, 854cm^{-1} indicates CH^2, 805 cm^{-1} indicates the aromatic compound. This indicates that functional group plays a major role in the synthesis of copper nanoparticle as they provide reducing groups which help in the synthesis of nanoparticles.

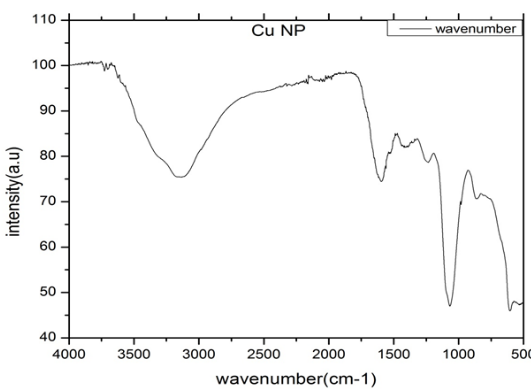

Fig.5 *FTIR spectroscopy* analyses of copper Nanoparticles

3) XRD Analysis

XRD pattern of synthesized copper nanoparticle using the leaf extract is shown in the fig. 6. The copper nanoparticle show high crystalline nature which corresponds the diffraction angle at 18º, 28º, and 32º. The average side of Cu Nps was calculated using Debye – Scherrer equation.

$D = k\lambda / \beta \cos\theta$

Where D is the Crystalline size of Nanoparticles, k is the Scherrer's constant ranges from 0.9. The size of the Cu NP in the extract was found to be 11 nm.

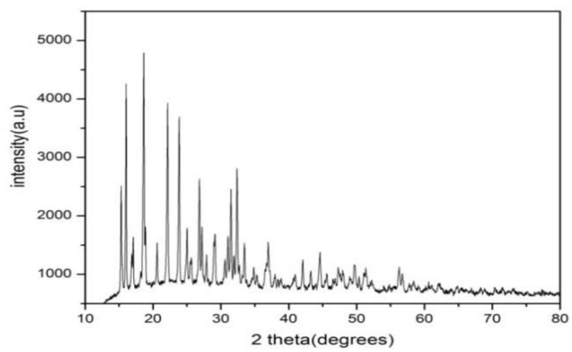

Fig. 6 XRD analysis of copper nanoparticle

Conclusion

From the above results we can able to conclude that, *Ocimum sanctum* extract can synthesis copper nanoparticle in an easy, less toxic, ecofriendly and cost effective manner. In this study very less amount of chemicals were used for the synthesis of copper nanoparticle and hence it is a green technology. The characterization of copper nanoparticle was done by UV-Visible spectrophotometer, FTIR, XRD analysis. The synthesized Cu NP will be used for further analysis of bioactivities they possess. Further studies are required using SEM for the structure analysis of synthesized copper nanoparticles and a comparison in structure and properties of nanoparticles synthesized at various temperatures.

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