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A Biological Approach for the Synthesis of Copper Oxide Nanoparticles by *Ixora Coccinea* Leaf Extract

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Abstract
Copper oxide nanoparticles have attracted huge attention due to catalytic, electric, optical, photonic, textile, nanofluid, and antibacterial activity depending on the size, shape, and neighboring medium. The use of plant leaves extract in the biosynthesis of nanostructured materials can be eco-friendly, non-toxic and cost effective approach. In this paper, we report a facile and green synthesis of copper oxide nano particles using *Ixora Coccinea* leaf extract. Highly stable copper oxide nanoparticles were synthesized by using *Ixora coccinea* leaf extract as a reducing agent as and copper chloride as a copper source at room temperature. Formation of copper oxide nanoparticles has been confirmed byUV-Vis absorption spectroscopy, X-ray diffraction (XRD), Dynamic light scattering analysis (DLS), zeta potentialstudy and Scanning Electron Microscope with the Energy Dispersive X-ray studies(EDX). Dynamic light scattering analysis shows average particle size of 167.1 nmwhereas high zeta potential value confirms the stability of formed copper oxide nanoparticles.The Scanning Electron Microscope reveals spherical morphology of nanoparticlesand Energy Dispersive X-ray analysis confirms the formation of highly purecopper oxide nanoparticles. The copper oxide nanoparticles from *Ixora coccinea* leaves areexpected to have applications in magnetic storage devices, solar energy transfer, sensors and super capacitors, Chemical plants etc. This neweco-friendly approach of synthesis is a novel, cheap, and convenient technique suitablefor large scale commercial production.

1. Introduction
Copper oxide nanostructures have attracted significant attention because of their wide range of applications such as high-Tc superconductors [1], sensors [2] and [3], catalytic[4], optical [5], electrical [6], giant magnet resistance materials [7] and [8] gas sensors [9],[10] and [11], solar energy transformation and preparation of organic inorganic nanostructure composites [12]. CuO is a p-type semiconductor with the band gap of ~1.7 eV [13]. Further it can be used as an antimicrobial, anti-biotic and anti-fungal agent when incorporated in coatings, plastics textiles, etc. [14]. Copper and copper-based compounds have efficient biocidal properties, which are generally used in pesticidal formulations [15] and several health related applications. Different methods available to prepare copper oxide nanoparticles namely sol–gel technique [16], Sonochemical [17], alkoxide based route [18], electrochemical methods [19], precipitation-pyrolysis [20], microwave irradiations [21], solid-state reaction method [22], thermal decomposition of precursor [23], etc. Chemical synthesis methods lead to presence of some toxic chemicals absorbed on the surface that may cause adverse effects in medical applications. Recently green synthesis of copper oxide nanoparticles by plants such as *Aloe barbadensis Miller* [24], latex *of Calotropis procera* L. [25] soybeans [26], *Manihot esculenta* [27], *Carica Papaya*[28], *Tridax procumbens*[29], *Magnolia kobus*[30], and *Gloriosa superba* [31] have been reported. In this regard using green methods in thesynthesis of copper oxide nanoparticles has increasingly become a need of time. Greenmethod using leaf extract of *Ixora coccinea* has been used for the first time for the synthesisof copper oxide nanoparticles.

Ixora coccinea Linn., (Rubiaceae) commonly known as jungle of geranium and red Ixora, is an evergreen shrub found throughout India. Depending on the medical condition, the flowers, leaves, roots, and the stem are used to treat various ailments in the Indian traditional system of medicine, the Ayurveda, and also in various folk medicines. The fruits, when fully ripe, are used as a dietary source. Phytochemical studies indicate that the plant contains important phytochemicals such as lupeol, ursolic acid, oleanolic acid, sitosterol, rutin, lecocyanadin, anthocyanins, proanthocyanidins, glycosides of kaempferol and quercetin. Pharmacological studies suggest that the plant possesses antioxidative, antibacterial, gastroprotective, hepatoprotective, antidiarrhoeal,

antinociceptive, antimutagenic, antineoplastic and chemopreventive effects, thus lending scientific support to the plant's ethnomedicinal uses.
The present investigation is an effort in this direction. So, the synthesis of Copper oxide nanoparticles has been reported so that *Ixora coccinea* could also be taken as potential candidate plant specimens for the synthesis of metal as well as oxide nanoparticles [32].

2. Experimental

2.1. *Materials*

Copper chloride dihydrate (99%purity) and sodium hydroxide (pellet 99%) were used as the introductory materials and supplied by Sigma-Aldrich chemicals. All glass wares were washed with sterile distilled water and dried in an oven before use.

2.2 *Methods*

2.2.1 *Preparation of the leaf extract*

Fresh leaves of *Ixora coccinea* plant were washed thoroughly with double distilled water.10 gm of leaves were grinded using mortar and pastel and it was extracted with 50 ml of distilled water. It was filtered through Whatman filter paper (no. 43) and total volume was made up to the 100 ml (i.e. 10% w/v of plant leaves extract) and used as stock solution for further studies.

2.2.2 *Synthesis of copper oxide Nanoparticles*

In a typical reaction mixture, 50 ml of aqueous 4mM copper chloride dihydrate ($CuCl_2.2H_2O$) was treated with 2 ml aqueous leaf extract of *Ixora coccinea* and stirred magnetically at room temperature, until the light blue colour changed to light green colour. Then the mixture is heated at 80°C for 2 hours. Afterwards, the mixture was treated with 1M sodium hydroxide drop by drop. As soon as, the sodium hydroxide comes in contact copper ions spontaneous change the green mixture to brown precipitate, indicating the formation of water soluble copper oxide nanoparticles. The concentrations of copper chloride solution and leaf extract were also varied from a 1 to 4 mM and 1% to 10% by volume, respectively. The brown precipitate was then taken out and washed repeatedly with deionized water followed by ethanol to remove the impurities for the final product. Then a brown powder was obtained after drying at 60°C in vacuum oven over night. The schematic producer was shown in figure 1.

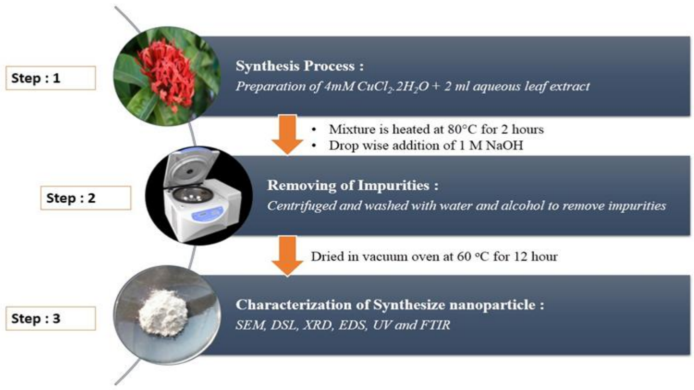

Figure 1: Graphical representation for formation of copper oxide nanoparticles.

2.3 *Characterization of copper oxide Nanoparticles*

2.3.1 *Uv-visible measurements*

Spectral analyses for the development of nanoparticles at different reaction conditions were observed using 3000+ UV–Vis spectrophotometer from 300 to 700 nm at a resolution of 1 nm at room temperature. Copper oxide nanoparticles gave sharp peak in the range of visible region of the electromagnetic spectrum.

2.3.2 *SEM-EDS analysis*

Scanning electron microscopy (SEM) analysis was performed with Qunta 200 FE SEM.

2.3.3. *XRD analysis*

The XRD analysis was carried out to determine structural changes. A normal focus diffractometer (Regaku Miniflex, Japan) source Cu target at 30 kV and 15 mA was used with scan rate of 3°/min. The data recorded in the range 2o -50o and analyzed using Jade 6.0 software.

2.3.4. *Particle size and Zeta potential measurements*
Particle size was determined by dynamic light scattering technique using Nano plus and zeta potential of copper oxide nanoparticle was measured using Nanoplus (Micromeritics, USA).

3. Results and discussion

3.1. *UV-vis analysis*
The biological approach for the formation of copper oxide nanoparticles using *Ixora coccinea* leaf extract was reported. Formation of copper oxide nanoparticles were confirmed by UV-VIS spectrophotometry. Fig.2 shows the UV–Vis absorption spectrum of copper oxide nanoparticles. The absorption spectrum was recorded for the sample in the range of 280–420 nm. The spectrum showed the absorbance peak at 360 nm corresponding to the characteristic band of copper oxide nanoparticles [33].

3.2. *X-ray diffraction Analysis*
The copper oxide nanoparticles biosynthesized from *Ixora coccinea* leaf extract were confirmed by the characteristic peaks observed in the XRD patterns, as shown in Fig. 3. The peak position with 2θ values of 35.54, 38.78, 48.78, 53.62, 58.4, 61.62, 66.28 68.1, 72.5 and 75.2°,which are in good agreement with those of powder copper oxide obtained from the International Centre of Diffraction Data card (JCPDS-80-1916) confirming the formation of a crystalline monoclinic structure. Well-defined and sharp copper oxide reflections in the observed XRD patterns verify the crystalline nature of copper oxide nanoparticles [34].

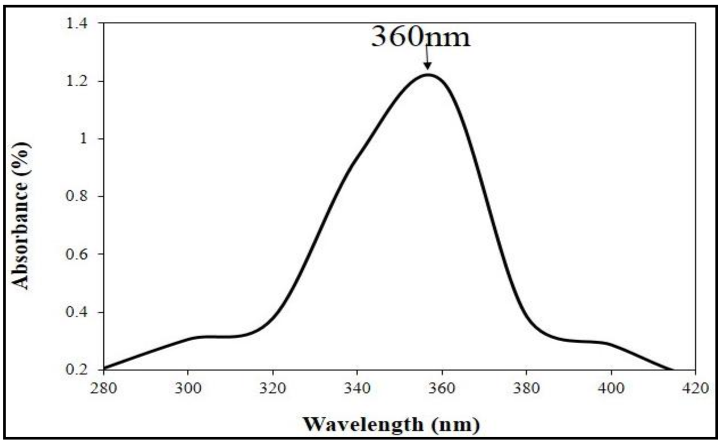

Figure 2:UV-visible spectrum of copper oxide nanoparticles synthesized.

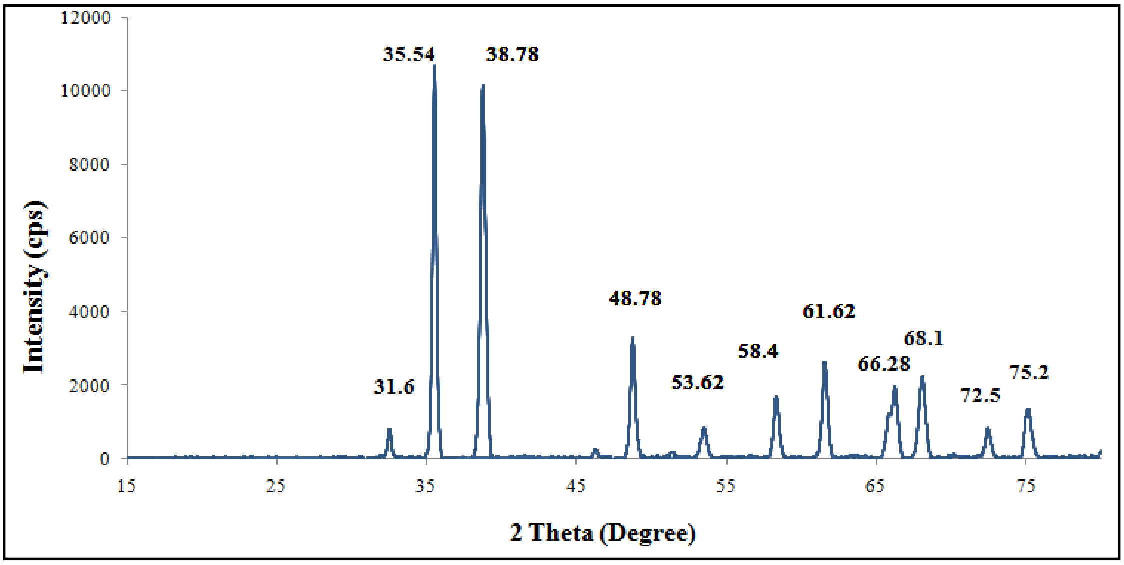

Figure 3: XRD pattern of synthesized copper oxide nanoparticles

3.3. *Dynamic light scattering (DLS) analysis*

Dynamic light scattering is a widely used technique for the determination of particle size in colloidal solution. The average size of the particles, size distribution, and polydispersity index (PDI) of the synthesized copper oxide nanoparticles were determined by this technique and the results are shown in Fig.4. It shows the average particle size of 167.1 nm and Polydispersity index is 0.345. The average particle size and PDI revealed that the produced copper oxide nanoparticles are monodispersed.

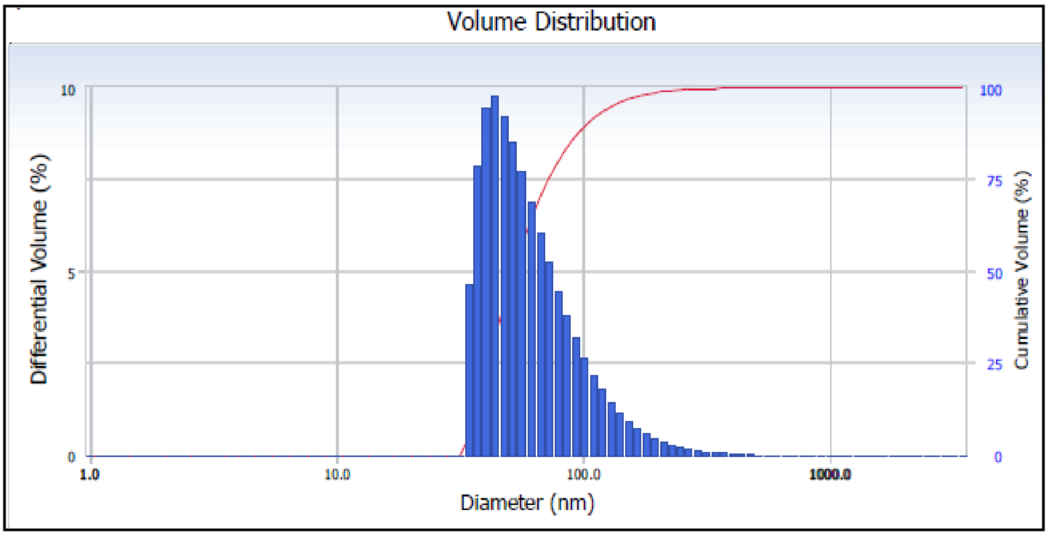

Figure 4: Particle size distribution of synthesized copper oxide nanoparticles.

3.4. *Zeta potential analysis*

Zeta potential analysis carried out to detect the surface charges acquired by silver nanoparticles, which can be used to gain further insights into the stability of the obtained colloidal silver nanoparticles. The magnitude of zeta potential gives an insinuation of potential stability of colloid. It should be noted that the particles with zeta potential values more positive than +30 mV or more negative than −30 mV are considered to be stable [35].

The electrostatic repulsive force between the nanoparticles depends on the charge which is present on the surface of the nanoparticle. When they are negatively charged, the process prevents the nanoparticles from agglomeration in the medium, leading to long-term stability. In the present study, the very high negative value of zeta potential confirms the repulsion among the particles and thereby increases the stability of the formulation. The zeta potential of the copper oxide nanoparticles was found to be −21.00 mV (Fig. 5). Based on the above results, it is concluded that the synthesized copper oxide nanoparticles are very stable [36].

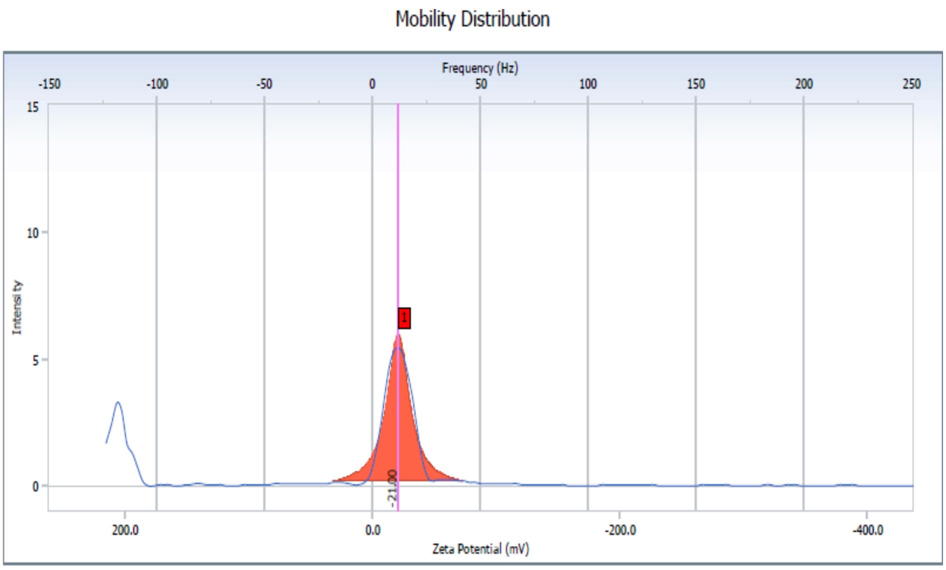

Figure 5: Zeta potential analysis of synthesized copper oxide nanoparticles.

3.5. *Energy dispersive X-ray Diffractive (EDX) analysis*

The Energy Dispersive X-ray Diffractive (EDX) study was carried out for the synthesized copper oxide nanoparticles to know about the elemental composition. EDX confirms the presence of copper and oxygen signals of copper oxide nanoparticle as shown in Table 1. The elemental analysis of the nanoparticle yielded 78.60% copper and 19.81% of oxygen which proves that the produced nanoparticle is in its highest purified form.

Table 1:EDX analysis of synthesized copper oxide nanoparticles

Element	Weight (%)
Cu K	78.60
O K	19.81
C K	1.51

3.6. *Scanning electron microscopy (SEM) Analysis*

The morphology of the prepared nanoparticles was examined using scanning electron microscopy. Fig. 6 shows the surface morphology of the copper oxide nanoparticles. SEM image showed individual copper oxide particles as well as a number of aggregates. The SEM image showed most of the nanoparticles are spherical in shape [37, 38].

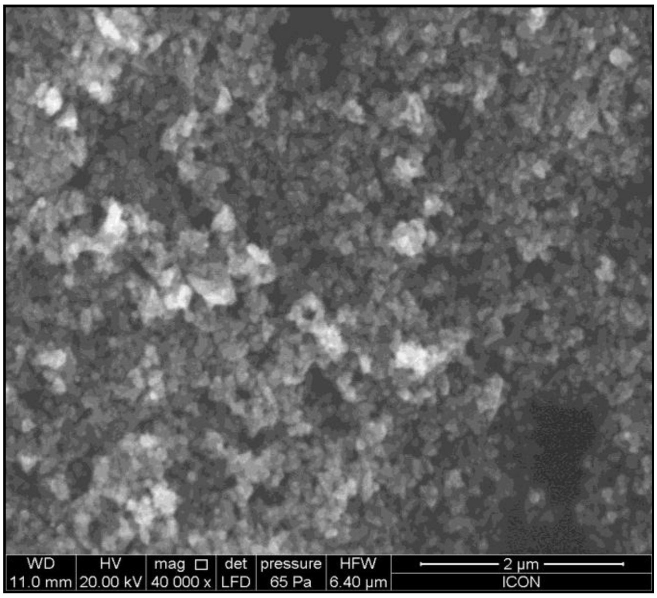

Figure 6: SEM image of the synthesized copper oxide nanoparticles.

Conclusions

The green chemistry approach used in the present work for the synthesis of copper oxide nanoparticles is simple, cost effective and the resultant nanoparticles are highly stable and reproducible. There is no report available for the synthesis of copper oxide nanoparticle by using any part of plant *Ixora cocinea*. The prepared copper oxide nanoparticles were spherical in shape and were characterized using XRD, EDX, DLS, UV-Vis absorption and SEM techniques. The SEM image showed that most of the nanoparticles are spherical in shape. X-ray diffraction confirms the formation of crystalline monoclinic structure. Particle size of 167.1 nm and polydispersity index of 0.345 was found, which indicates there is uniform size distribution of nanoparticles. The zeta potential was found to be −21.00 mV. The high value confirms the stability of copper oxide nanoparticles. Copper oxide nanoparticles prepared from above mentioned route are expected to have more extensive applications such as efficient antimicrobacterial agent, chemical sensors, and doping materials in semiconductors etc. This process is an economical method for the preparation of nanocrystalline copper oxide with respect to energy, time and simplicity and can be used for large scale synthesis of copper oxide nano particles. This green method of synthesizing copper oxide nanoparticles could also be extended to fabricate other, industrially important metal oxides.

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