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Preparation and Characterization of Copper Oxide Nanoparticle-Determination of Its Structural and Optical Properties.

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Abstract A facile, innovative and affordable green route has been demonstrated for the formation of Copper oxide nanoparticles by biogenic method using aqueous leaf extract of Calotropis Gigantea which acts as a reducing and stabilizing agent. Copper nanoparticles received much attention owing to its high electrical conductivity, high liquefaction point, low electrochemical migration behavior and low cost.The property of copper nanoparticles mainly depends on the synthesis route and their process parameters. The prepared Copper oxide nanoparticles were characterized by a host of different techniques such as X-ray diffraction (XRD), Diffuse reflectance spectroscopy (DRS), Zeta potential and Fourier transform infrared spectroscopy (FT-IR). The XRD pattern confesses that Copper oxide nanoparticles associate to orthorhombic structure. The DRS absorption spectrum shows band gap energy corresponds to wide band gap semiconductor The FT-IR spectra indicate the presence of chemicals which are responsible for biochemical reaction. The Zeta Potential value revealed the stability of Copper oxide nanoparticles

Keywords-Calotropis gigantean leaf, XRD,FTIR, DRS, Zeta Potential.

INTRODUCTION

Nature developed “nanotechnologies” over billions of years, employing enzymes and catalysts to arrange with exquisite precision different types of atoms and molecules into complex microscopic structures that make life possible The physical and chemical properties of nanostructures are distinctly different from those of 1 atom (molecule) and bulk matter with a similar chemical composition. Within the past decades, many efforts are made within the synthesis of metal nanoparticles owing to their unusual properties and potential applications in optical, electronic, catalytic, and magnetic materials so on [1]. CuO metal nanoparticles attracted considerable attention due to their catalytic, optical, and conducting properties. Their synthesis has been achieved via various routes including radiation methods, micro emulsion techniques, supercritical techniques, thermal reduction, laser ablation, metal vapor synthesis, chemical reduction, green method etc [2]. Using green leaf as reducing agents to organize CuO nanoparticles in aqueous solutions is attractive because organic solvents aren't used and thus the corresponding pollutants are absent. Metal nanoparticles have received much interest in recent time both from academia and industry. Utilization of environmentally benign, cheap and simply available supports/stabilizers for the syntheses of metal nanoparticles in context of green science are the realm of cutting edge research. It’s therefore important to synthesize and characterize supported metal nanoparticles and to see their reactivities for the event of novel synthetic methodologies in organic synthesis. On the other hand, development of cost effective and greener methodology for the synthesis of specialty chemicals is that the area of immense possibilities and tremendous importance within the captive global markets. During this work, the preparation of CuO nanoparticles by biogenic method using aqueous leaf extract of Calotropis Gigantea which acts as a reducing and stabilizing agent. The resultant nanoparticles were characterized by X-ray diffraction (XRD), Diffuse reflectance spectroscopy (DRS), Zeta Potential and Fourier transform infrared spectroscopy (FT-IR).

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MATERIALS AND METHODS

Calotropis Gigantea leaf was collected from our institution campus. Metallic reagent AR grade Copper sulphate ($CuSo_4$.$5H_2O$) precursor was purchased from scientific suppliers.

Preparation of Calotropis Gigantea Leaf Extract

The Leaf of Calotropis Gigantea were washed for several times and later with distilled water. They were grinded with the help of a mixer grinder and then the extract was taken from the grinded paste. It was then filtered and used for the synthesis process.

Synthesis of CuO NPS

Copper sulphate ($CuSo_4$.$5H_2O$) solution of 0.2 M was prepared using double distilled water. The prepared leaf extract of Calotropis Gigantea was added drop wise into the $CuSo_4$.$5H_2O$ solution until the precipitate forms. The solution was stirred continuously for four hours. The observed color change from blue cooler to pale green (fig.1) Indicates the reduction of copper ions and the formation of copper oxide nanoparticles and dried in the hot air oven for one day at 100°C,the dried CuO NPs particles are grinded and calcinied at 500°c for 3 hour . All the dried CuONPs were made into a fine powder with the help of a mortar and were used for further characterization to identify the various properties of CuO Nanoparticles.

Figure 1: Reaction process using Calotropis Gigantea reducing agent

XRD Analysis

XRD pattern of CuO NPs was shown in Fig. 2. XRD characterization was performed to determine the crystallinity and the typical peak of CuO NPs diffractogram by comparing the diffraction angle 2θ to the literature (JCPD Number 771898). Based on XRD pattern, there were some peaks appeared 2θ values at 13.73˚, 16.43˚, 18.71˚, 22.73˚, 23.93˚, 25.70˚, 27.88˚, 29.24˚, 31.85˚, 32.53˚,33.37˚, 35.55˚, 36.45˚and 43.31˚. The data have conformity with the JCPDS data (771898) of CuO, indicating that CuO NPs have been successfully synthesized. Miller indices of synthesized CuO NPs were (0 1 2), (0 2 0), (2 0 0), (2 1 2), (0 1 4), (0 3 2), (3 1 1), (1 3 3), (3 0 3), (2 2 4), (1 4 1), (3 2 3) and (4 0 4) with orthorhombic crystalline phase. The average crystalline size (D) is calculated by Debye-Scherrer's relation[3].

$$D=K\lambda/\beta COS\theta$$

Where k is the Scherer constant, accounts for the factor. K is 0.9λ is the wavelength of X-ray,
β is the " full width at half maximum(FWHM) in radians of the X-ray diffraction peak
θ is the Bragg's angle.
The average crystalline size (D) has been calculated is 38 nm.

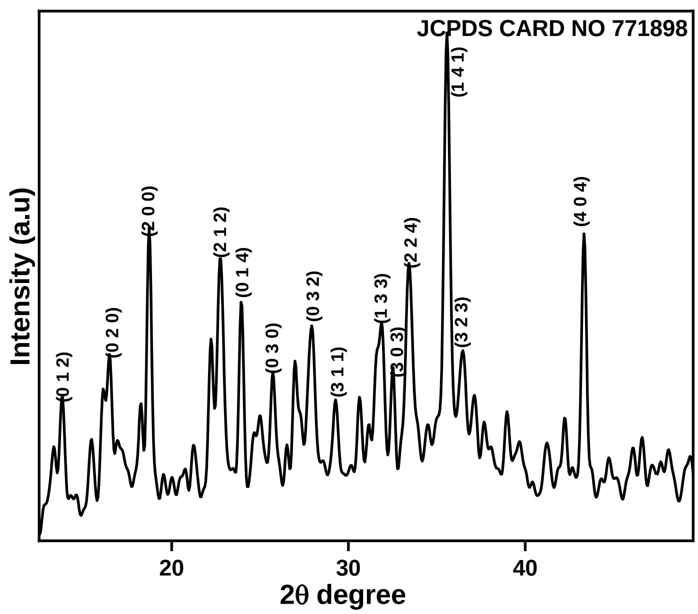

Figure 2: XRD pattern of CuO NPs

By using Williamson- Hall analysis (W-H), crystal sizes along with strain associated due to lattice dislocation can be determined from XRD data shown in figure 3. The proposal efficient method for separation of strain and size effects on broadening is watching the height width as a function of diffracting angle 2θ. The modified W-H equation is expressed as follows[4],

$$\beta \cos\theta = \left(\frac{4\lambda}{D}\right) + (4\varepsilon \sin\theta)$$

where, D is the average crystalline size and ε is the strain and assumed to be uniform in all crystallographic directions. The above equation is within the sort of line equation where the term βcosθ was plotted against 4sinθ for the well-liked orientation peaks of the prepared samples. From uniform and Stress deformation model the strain and slope are extracted[5]. Accordingly, the slope and y-intercept of the fitted line represent strain and particle size, respectively. The average size of synthesized nanoparticles is found to be (D) =38 nm.

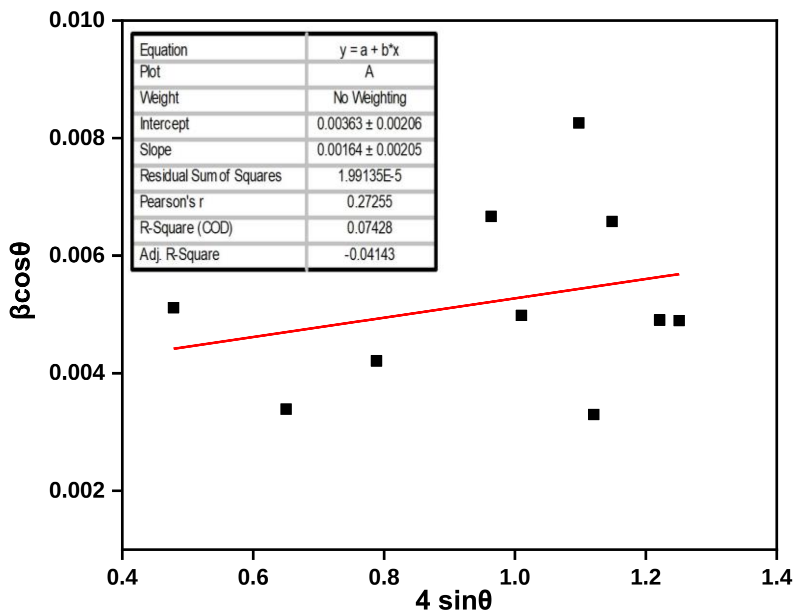

Figure 3: Williamson Hall plot forCuO NPs

FTIR Analysis

FTIR characterization was performed to determine the functional groups in CuO NPs as shown in Fig.4.Chemical Composition of the leaf Calotropis Gigantea include Cardenolides, Steroids, Tannins etc. The FTIR ranges of those compounds are confirmed by the graphs. The vibration of Cu-O bond at frequency 596 cm-1[6,7]. The wide and robust absorption appeared at 3072 cm-1 as O-H group of alcohol and 2367 cm-1 as C-C alkane groups, while there have been peaks at 1635 cm-1 as C=C stretching, 1423 cm-1 as C-H bend alkane , and 1100 cm-1 as C-N group, indicating the role of Calotropis Gigantea leaf in the process of CuO NPs formation.

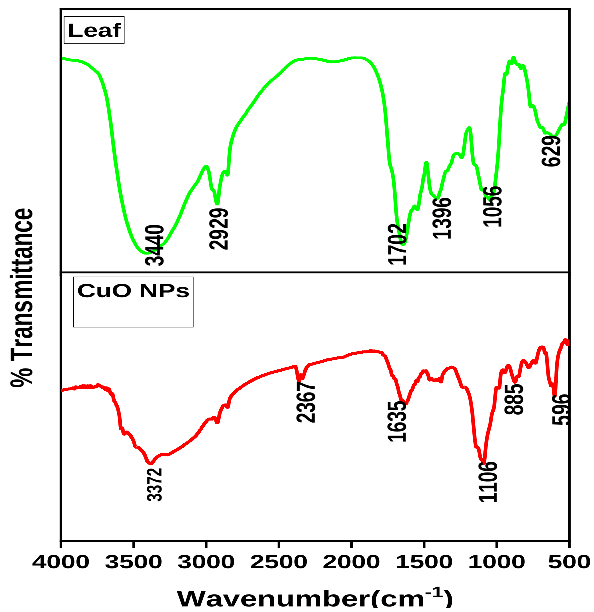

Figure 4: FTIR spectra of Leaf and CuO NPs.

UV-Vis DRS Analysis

The UV-Vis DRS characterization was performed to measure the energy band gap of CuO NPs as shown in Fig is synthesized by using Calotropis Gigantea leaf extract. The sample has a clear and strongly observed radiation peak below at 417 nm. The band gap energy (Eg) of CuO was obtained from the wavelength value corresponding to the intersection point of the vertical and horizontal part of the spectrum is 2.9 eV.

The reflectance spectra is also analyzed using the Kubelka- Munk relation . To convert the reflectance data into a Kubelka-Munk function [8-11](equivalent to the absorption coefficient) F (R), the subsequent relation was used

$$F(R) = \frac{(1-R)^2}{2R}$$

Where, R is the reflectance value. Band gap energy of the sample was estimated from the variation of the Kubelka-Munk function with photon energy. Fig.4 shows the Kubelka-Munk plots for the CuO NPs .It is used to determine their band gap energy associated with their direct transitions. The CuO exhibits direct band gap energy is 2.9 eV which has the characteristics of Wide-band gap semiconductor materials have the ability to operate at higher temperatures.

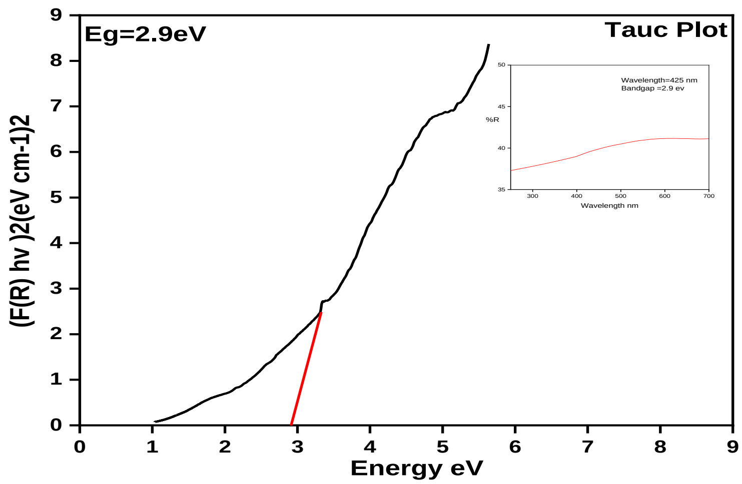

Figure 5: Tauc's plot of CuO NPs

Zeta Potential

When nanoparticles are dispersed in water, the particles acquire charge upon dispersion they have a tendency to aggregate because of Van der Waal's forces of attraction. The aggregation is also prevented if the electrostatic repulsive forces overcome attractive Van der Waal's forces. Zeta potential is taken into account as a measure of charges on the nanoparticles' surface, the higher magnitude of which improves dispersion stability of nanoparticles. The zeta potential of oxide nanoparticles prepared using extract of Calotropis Gigantea leaf well dispersed in water was found to be 7.33mV with relative ease, suggesting its application for preparation of nanofluids shown in Figure 8 .The Zeta Potential gives a sign of the charge present on the particles surface. The positive value implies that CuO nano particles are positive charged. The zeta potential becomes more positive when decreasing the pH[12,13]. It means on the surface of nanoparticles encompasses a net charge so the CuO nanoparticles have superficial groups with charge and indicate the stable suspension on water.

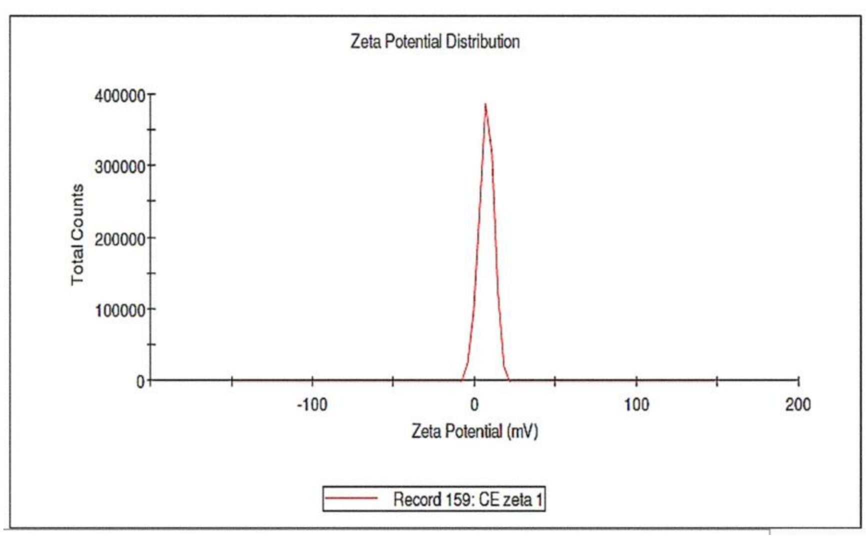

Figure 6: Zeta Potential Diffusion for CuO NPs

CONCLUSION

Copper oxide nanoparticles (CuO NPs) have been successfully prepared through leaf extract of Calotropis Gigantea by green method. The particle size and structure of synthesized nanoparticles were determined from the XRD data.The FTIR spectrarevealed the functional groups of stretching bands for CuO NPs. UV-Vis DRS analysis confirmed the synthesized CuO NPs as wide band gap semiconductor with the energy band gap of 2.9 eV, Zeta potential confirmsthe positive stability CuOnanoparticles. Therefore, the present research was designed to prepared eco-friendly, stable and nontoxic CuO nanoparticles from the Calotropis Gigantea leaf extract.

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