SYNTHESIS OF COPPER OXIDE NANOPARTICLES BY CO-PRECIPITATION METHOD USING DIFFERENT CONCENTRATIONS OF PRECURSOR AT DIFFERENT pH VALUES

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ABSTRACT:

Copper oxide nanoparticles were synthesized via the coprecipitation method using different concentrations of copper nitrate with varying pH values. The relevant properties of synthesized copper oxide nanoparticles were studied with the help of SEM, XRD,and FTIR. The XRD peaks with the JCPDS cards confirmed the formation of copper oxide nanoparticles and also estimated their average crystalline size in the nanometre range. SEM images predicted that the nanoparticles are nano rod-shaped, uniformly distributed, andcrystalline. The Optical properties are studied by UV- Visible spectroscopic techniques. The effect of pH and concentration on the optical properties of nanoparticles has been discussed in detail.

Keyword: Nanoparticles, optical property, spectroscopy.

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1. Introduction

Nanoparticles have gained large attention, especially in research due to their distinct optical, magnetic, chemical, and physical characteristics as compared to their bulk material. Interest in metal oxide nanoparticles has been increased because they can be widely used as catalysts (*Jianliang et al. 2011*), sensors (*Yang et al.2007*), antimicrobial *(pit so et al.2021*)applications, and many more. One of the prominent metal oxides is copper oxide because of itsamazing antimicrobial and biocidal properties. “Copper oxide” is considered one of the good semiconductor metals with its unique electrical, magnetic and optical properties (*Phiwdang etal*). On the other hand, having these traits is used in the making of different sensors, which can be used in air quality detection, security, defense, and health (*Jhansi et al. 2016)*. This black powdery substance is often implemented for therapeutic uses for its glucose sensing and dopamine-sensing abilities (*Verma and Kumar, 2019*). The small size of these substances results in them being implemented at commercial levels, thereby resulting in them often being synthesized using precipitation methods.

In the given work, the major aim, to synthesize copper(ii)oxide nanoparticles by coprecipitation method and to investigate the impact of pH and concentration of precursor on optical properties of copper oxide nanoparticles have been discussed. The synthesized nanoparticles were analyzed by SEM, X-ray diffraction, and FTIR spectroscopy.

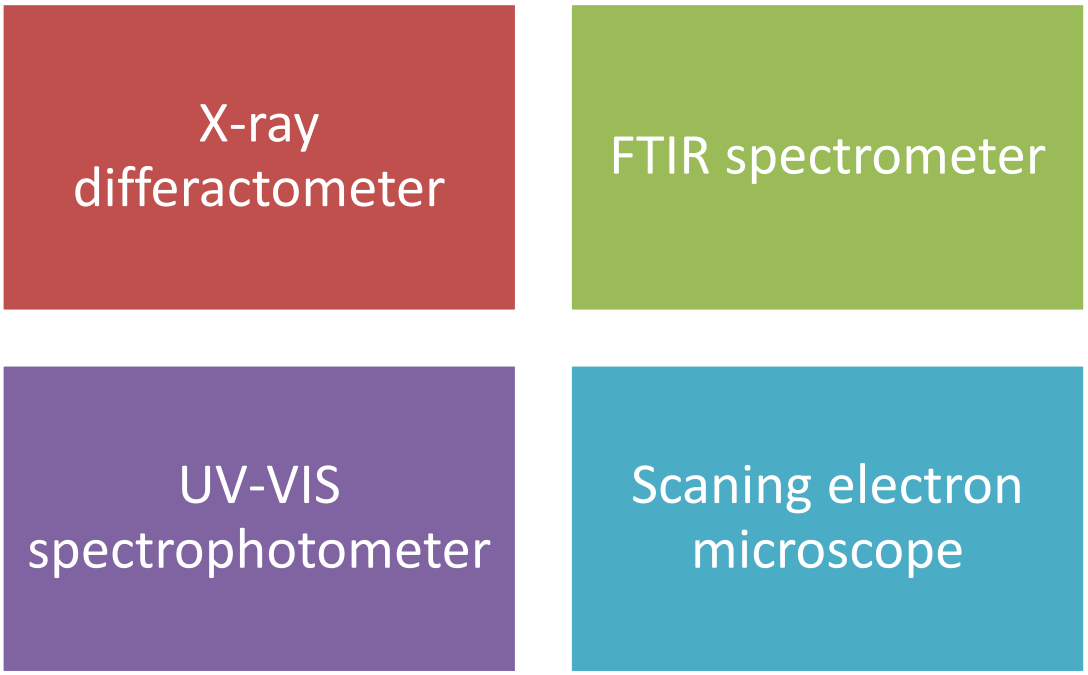
2. Experimental Details

2.1 Materials

The study was conducted by implementing an experimental method. The use of the following chemicals has been done to conduct this experiment.

$Cu(No3)2.3H2O$, 1M NaOH solution, absolute $C2H5OH$, distilled water. All the materials being used here are of A.R grade during the experiment.

2.2 Instrumentation



The appropriate instruments are required for carrying out the "experimental study". This experiment involves the use of the following instruments:

The use of ***"X-ray diffractometry"*** has been done to analyze the structural configuration and crystal size of CuO nanoparticles. The measurements obtained with the help of XRD have beenused for crystal analysis of CuO nanoparticles. XRD measurements were taken with a Rigakumini flex II diffractometer in the 2θ range of 20^{o} - 60^{o} using Kα radiation of the wavelength λ =1.5406. The size of the particle, its distribution, and its morphology have been investigated with the help of ***"Scanning electron microscopy"*** SEM. The use of JEOLJSM-6510 (Japan) SEM has been used in the experiment.

The information about optical studies has been gained with the help of ***"diffuse reflectance spectrum"*** which was recorded with Shimadzu UV-visible 3600 plus spectrophotometer. Thishas been furnished with a sphere accessory in the 200-500 nm range. All the optical analysis has been done at room temperature under ambient conditions.

2.3 Synthesis of CuO Nanoparticles with the help of co-precipitation method

The following procedure had been followed to conduct the experiment:(*K.Jhansi et al2016*). CuO nanoparticles were synthesized by the coprecipitation method using Cu (No3)2.3H2O as a precursor. 50 ml distilled water was used to dissolve 0.5 M cupric nitrate andthe solution was stirred on a magnetic stirrer for 15 minutes. 1M of NaOH solution was addedto the above solution dropwise until the pH reached 10. The above solution was then stirred magnetically for 2:30hr till the blackish-brown precipitate of Cu(ii) O nanoparticles have beenobtained. The precipitate was then washed with the help of deionized water and ethanol so thatexcess impurities drained out. The precipitate was left to be dried to get its powdered form. The black powdered CuO nanoparticles were annealed at 450^{o}C for an hour in order to achieve ***"stabilized CuO nanoparticles".*** The same set experiment was repeated for pH 8 and pH 12 also. The above process was repeated for different concentrations of precursor (1M, 1.5M).

3. Result and Discussion

3.1 Physical characterization

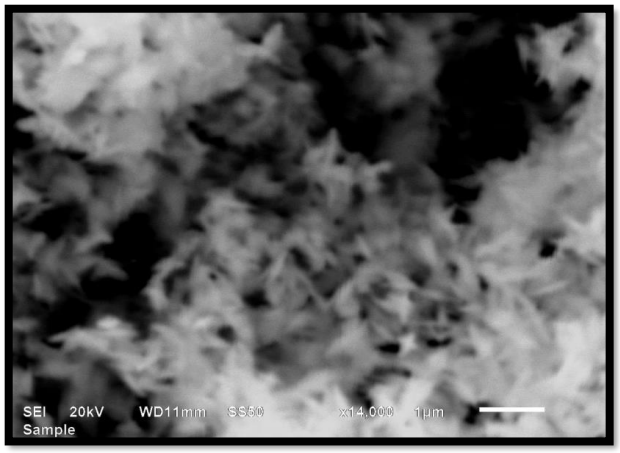


Figure 1: Structure of CuO nanoparticles obtained from SEM image (14000x)

Fig.(1-2) illustrated the SEM images of CuO nanoparticles revealing the morphology, and shape of CuO nanoparticles synthesized by the coprecipitation method. SEM images (fig.1) investigated through the magnification of 1µm showed nano rod-like structure of CuO nanoparticles. In fig.2 SEM images magnification of 5µm showed an almost uniform distribution of particles with somewhat agglomeration seen.

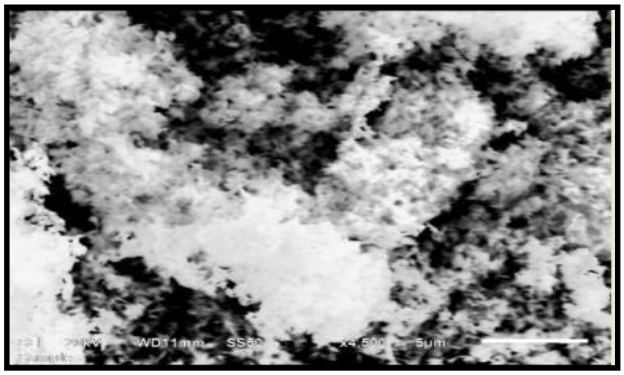


Figure 2: Structure of CuO nanoparticles obtained from SEM image (4500x)

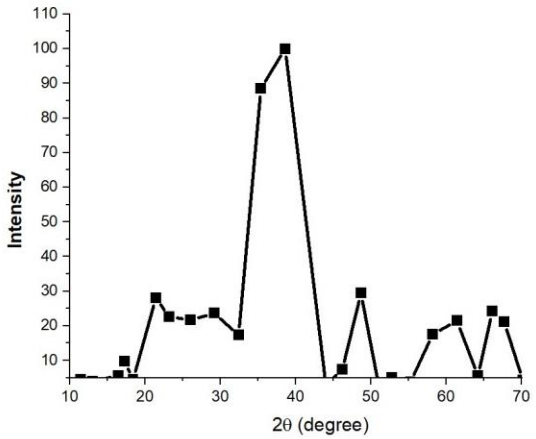


Figure 3. XRD Pattern of CuO nanoparticles

XRD pattern of as-synthesized Cu(ii)O nanoparticles at concentration 0.5 M at pH 10 is shown in figure (3). The XRD peaks with JCPDS cards confirmed the production of copper oxidein the monoclinic phase. The characteristic peaks located at 2θ =38.97̊ ,48.74˚, 61.5˚, 68˚ are assigned to (111), (-2 0 2), (-113), (-2 2 0) plane of orientation of CuO respectively (JCPDS 80-1268).

The particle size of CuO was estimated by using the formula ***"Debye- Scherer- formula".***

$$D = (0.89\lambda/\beta \cos\theta)$$

In the above expression, the size of the particle is denoted by D, λ is the X-ray wavelength, thewidth of the full peak is denoted by β, and the angle of Bragg diffraction is depicted by θ (Vyas *et al.* 2021). Using the above formula, the size of the nanocrystal was found to be ***8.6nm.*** As per the above graphical representation and the calculated size of the nanoparticles, it has been observed that the sample does not contain any impurities and is highly stable.

3.2 FTIR SPECTROSCOPY:

The main principle of FTIR helps us in identifying the functional group present in the compound. The FTIR analysis was carried out by scanning in a range of 600-4000 cm^{-1}. Thecharacterized peak in the range from 600-3413cm^{-1} was more visible

for synthesized nanometal oxides. (*Pitso T.el al2021*). Figure (4) shows the FTIR spectra of powdered copper oxide nanoparticles. The peaks at 640 cm^{-1} indicate the formation of the monoclinic phase of pure CuO (*Nogueira et al.2016*). The band obtained at 1643 cm^{-1} show the carbonyl C=O stretching bond. A Strong broad band observed at 3556-3310 cm^{-1} is attributed to the bending and stretching vibration of absorbed water and the surface hydroxyl group *(Dhinesh babu et al.2016*).Peaks at 1366cm^{-1} were assigned to C-H stretching bond.

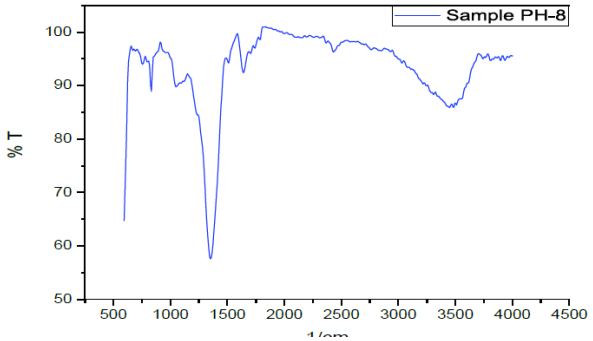


Figure 4. FTIR spectra of copper oxide

3.3 Optical characterization

The optical characterization of the CuO nanoparticles has been done by absorption spectra at different pH and concentrations (fig. 5,6). The absorption spectra study of CuO nanoparticles shows a sharp peak at 220 nm for 1.5 M due to the direct transition of electrons. The absorptionband was at 230 for 1M and 240 nm for 0.5 M concentration shown in figure 5. This adsorption peak showed that as concentration decrease absorption edge shift toward a longer wavelength or we can say as the concentration increase band gap increase. The increase in the band gap isbecause of the quantum confinement effect (*Prakash et al.2013*).

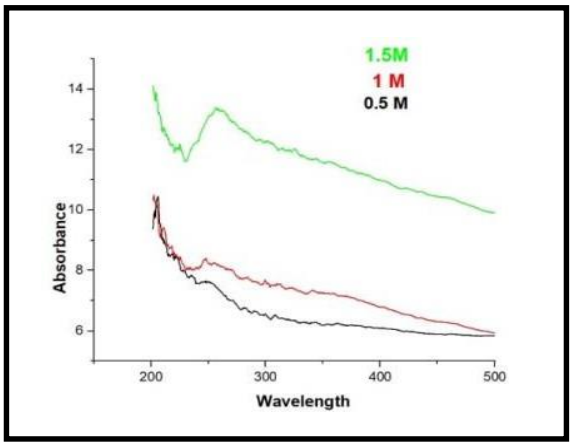


Figure 5: Absorbance of CuO nanoparticles at different concentrations

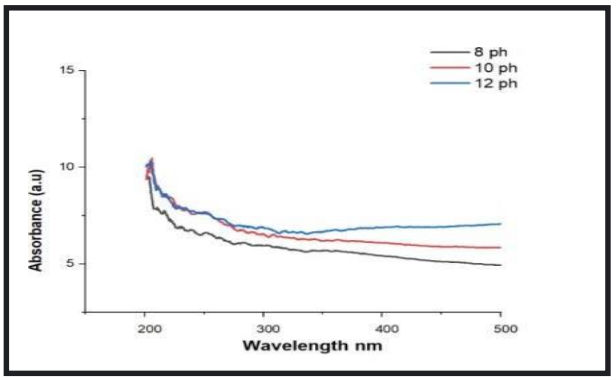


Figure 6: absorption spectrum of CuO nanoparticles at distinct pH

The above figure 6 graphically represents the absorption spectra of the CuO nanoparticles in distinct pH. As the pH increased particle size decreased, and the surface-to-volume ratio increased with a decrease in particle size. Due to this effect surface atom has low atomic interaction which leads to reduced conduction band energy and increased valance band energy which results in increased band gap (*Prakash et al.2013*). A larger band gap means electrons required more energy to excite from the valance band to the conduction band so required lightof higher frequency or lower wavelength.

4. Conclusion

Copper oxide nanoparticles were synthesized by co-precipitation method using different concentrations of precursor (0.5M, 1M,1.5M cupric chloride) at different PH (8,10,12). X-Ray Diffraction, SEM, FTIR, and UV- VIS

characterization justified well with the formation of CuO nanoparticles. SEM images confirmed that the morphology of CuO nanoparticles is nano rod-shaped with uniform distribution. UV-VIS spectra clearly indicate the decrease in particle sizewith the increase in concentration and pH, which may be attributed to the quantum confinementeffect.

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