International Journal of Scientific Research in Physics and Applied Sciences
Vol.6, Issue.2, pp.30-34 April (2018)

Research Paper

E-ISSN: 2348-3423

Analysis of Structural and Optical Features of CuO Nanoparticles Synthesized at Different Molarities

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Available online at: www.isroset.org

Received: 16/Mar/2018, Revised: 22/Mar/2018, Accepted: 14/Apr/2018, Online: 30/Apr/ 2018

***Abstract*—** The sol-gel method has been implemented to synthesize CuO nanoparticles at 100^0C.The monoclinic phase structure of CuO nanoparticles has been confirmed by X-ray Diffraction (XRD) pattern. Crystal size and dislocation density of the nanoparticles have been estimated from XRD data. Optical band gaps have been calculated from optical absorption spectra. The chemical composition of the nanoparticles has been investigated with using fourier transform infra red (FTIR) spectroscopy. The crystal surfaces of the nanoparticles have been investigated by scanning electron microscopy (SEM).

Keywords*—Nanoparticles, XRD, optical band gap, FTIR, SEM*

I. INTRODUCTION

Nanoparticles differ from bulk materials due to their peculiar electronic, optical and chemical features. Copper oxide (CuO) nanoparticles are extensively utilized in plenty of applications such as gas sensor, photo catalyst, antimicrobial, solar cells, humidity sensor, toxic element remover from water [1-13]. Numerous methods are used to synthesized CuO nanoparticles such as chemical bath deposition, vapour deposition, microwave assist synthesis, hydrothermal synthesis, sol-gel, spray pyrolysis solid state thermal decomposition [14-20]. The sol-gel method is one of the cost-effective methods for preparing CuO nanoparticles. In this paper, we have prepared CuO nanoparticles with the help of sol-gel method and analysed its different structural and optical features.

This paper has been arranged into four segments - Introduction, Materials and Methods, Results and Discussion and Conclusion. Introduction segment gives the brief review of literature related to our present work. Materials and Method segment contains the materials used for the preparation of CuO nanoparticles and details of preparation procedure of CuO nanoparticles at different molarities by sol-gel method with special reference to characterization tools. Results and Discussion segment provides a brief detail on the characterizations of the CuO nanoparticles and analysis and discussion of the results obtained. Conclusion segment provides major conclusions drawn from the results.

II. MATERIALS AND METHODS

All the required materials for synthesizing CuO nanoparticles by the sol-gel method, such as $CuCl_2.2H_2O$, NaOH and glacial acetic acid were bought from the market with the highest purity. The aqueous solution of $CuCl_2.2H_2O$ (0.2M) was prepared in 50ml DI water dissolving the required amount of $CuCl_2.2H_2O$ in a beaker. Dropwise addition of 1 ml of glacial acetic acid was done to the above solution with constant stirring and then heated to 100^oC. The colour of the aqueous solution turned from blue to green. 8.5 pH was maintained by dropwise addition of 8M NaOH solution to the above solution. A change in colour of the solution was observed and a black precipitate was formed. The precipitate was filtered and washed 4 times with deionized water. Then the precipitate was dried in air and converted into powder using mortar. The powders were used for further analysis and characterization of CuO nanoparticles.

A similar method was applied to synthesized CuO nanoparticles from 0.4M and 0.6M $CuCl_2.2H_2O$ aqueous solution.

Structural characterization of prepared CuO nanoparticles were done by XRD using Philips X'pert Diffractometer with CuKα radiation (λ=1.5406 Å). Carry 300 scan UV-Visible spectrophotometer was used to measure optical absorption spectra. Fourier Transform infra red (FTIR) spectra of CuO nanoparticles were obtained from SHIMADZU in the wave length range 400-4000cm^{-1}. The morphology of prepared

CuO nanoparticles was studied by Scanning electron microscope, ZEISS, SIGMA.

III. RESULTS AND DISCUSSION

A. Structural Analysis

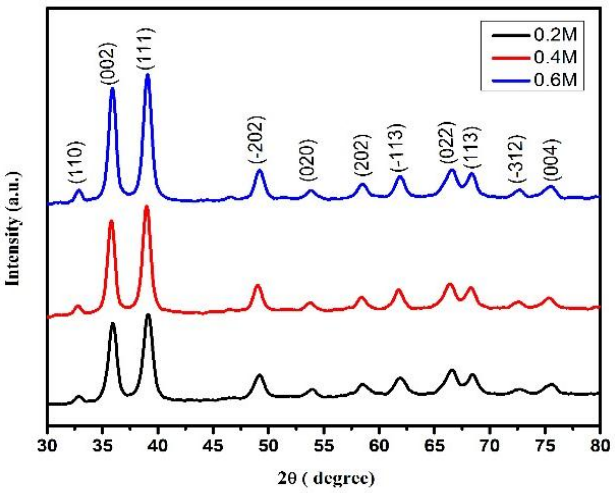


Fig 1: XRD pattern of CuO nanoparticles prepaped at 0.2M, 0.4M and 0.6M

X-Ray Diffraction pattern is used for determining the structure and phase of prepared CuO nanoparticles. Fig.1 shows the XRD diffraction pattern of prepared CuO nanoparticles at different molarities (0.2M, 0.4M and 0.6M). The diffraction pattern exhibits clear peaks centred at various angles corresponds to (110), (002), (111), (-202), (020), (202), (-113), (022), (113), (-312) and (004) planes as shown in Table1. All the peaks in the diffraction pattern show the prepared CuO nanoparticles are of monocline structure. The peaks are matched with the standard data of ICDD card no. 89-5895. The Debye-Scherrer's equation is used to find the crystallite size (D) of prepared samples [21].

$$D = \frac{K\lambda}{\beta \cos\theta} \qquad (1)$$

Where K is a constant and equal to 0.9, λ = wavelength of X-ray radiation (λ=1.54056 Å for CuK_α radiation), β = FWHM (full width at half maximum, in radian) and θ =the Bragg's diffraction angle. The calculated values of crystallite size are given in Table 1.

The dislocation density of CuO nanoparticles has been estimated by using the equation [22]

$$\text{Dislocation Density } (\delta) = 1/(D^2) \qquad (2)$$

Where D is the crystallite size. The calculated values of dislocation density of the CuO nanoparticle samples are given in Table 1.

Table1.Structural parameters of CuO nanoparticles prepared at various molarity

Molarity	Angle	hkl	Size obtained by Debye Scherrer formula	Dislocation Density
	($2\Theta^0$)		(nm)	($\delta x10^{16}$ m^2)
CuO(0.2M)	32.86	(110)	10.99138	0.828388
	35.86	(002)	9.583741	1.089603
	39.21	(111)	9.679324	1.06819
	49.24	(-202)	7.577513	1.74295
	53.99	(020)	9.566095	1.093626
	58.99	(202)	7.145324	1.960173
	61.96	(-113)	8.040692	1.547931
	66.58	(022)	8.246726	1.47155
	68.53	(113)	7.524922	1.767398
CuO(0.4M)	32.89	(110)	11.91257	0.705224
	35.89	(002)	10.79271	0.859166
	39.09	(111)	9.668031	1.070686
	49.24	(-202)	8.659733	1.334533
	53.87	(020)	7.417666	1.818879
	58.56	(202)	9.035591	1.225816
	61.94	(-113)	9.911643	1.018702
	66.57	(022)	7.530069	1.764983
	68.36	(113)	8.985342	1.239565
CuO(0.6M)	32.74	(110)	12.43965	0.646729
	35.85	(002)	11.34733	0.777232
	39.1	(111)	10.02501	0.995793
	49.1	(-202)	10.0241	0.995972
	53.85	(020)	8.506046	1.383193
	58.4	(202)	8.616924	1.347826
	61.71	(-113)	9.838855	1.03383
	66.4	(022)	8.176828	1.496817
	68.25	(113)	9.254641	1.168474

B. Optical Absorption Analysis

Different optical properties of the prepared CuO nanoparticles were studied by recording optical absorbance spectra in the range 200-800nm. The information of optical absorbance and band gap energy of a material predicts the area in which it can be used.

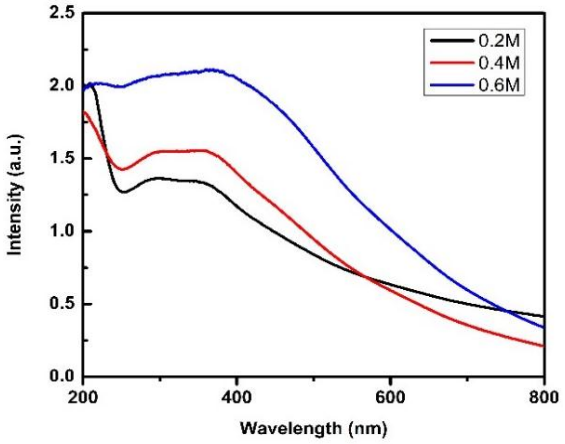


Fig 2. Absorption spectra of CuO nanoparticles prepared at 0.2M, 0.4M and 0.6M

The Fig 2. shows optical absorption spectra of CuO nanoparticles synthesized at different molarities (0.2m, 0.4M and 0.6M) [23-24].

The optical band gap energy (E_g) is calculated by using Tauc's formula [25]

$$(\alpha h\nu)^{1/n} = A(h\nu - Eg) \qquad (3)$$

Where α is the absorption co-efficient, $h\nu$ is the incident photon energy, A is a constant. For direct band gap material, n=1/2. The band gap energy has been estimated by plotting graph $(\alpha h\nu)^2$ versus $h\nu$ and then extrapolating the linear region of plots as shown in Fig 3. The band gap energy of CuO nanoparticles prepared at 0.2M, 0.4M, 0.6M are estimated as 4.94, 4.48 and 2.76 eV respectively.

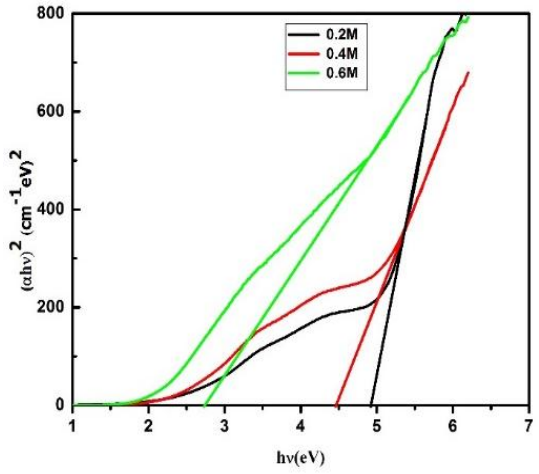


Fig 3. $(\alpha h\nu)^2$ vs $h\nu$ spectra of CuO nanoparticles prepared at 0.2M, 0.4M and 0.6M

C. Fourier Transform Infrared (FTIR) Analysis

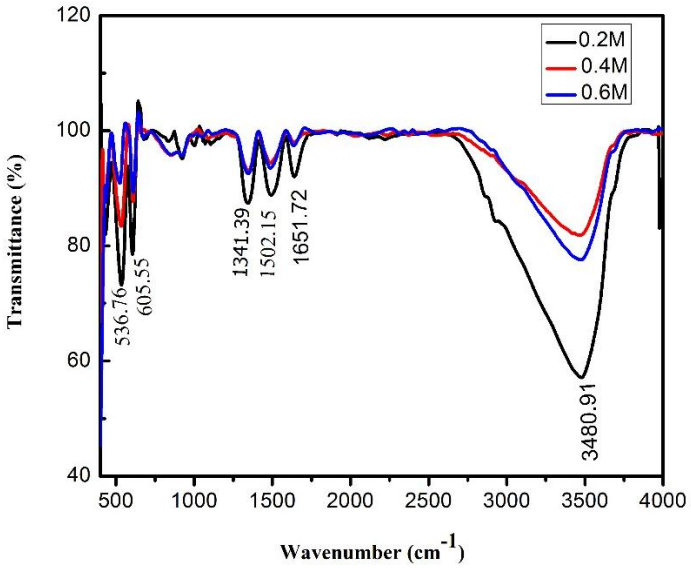
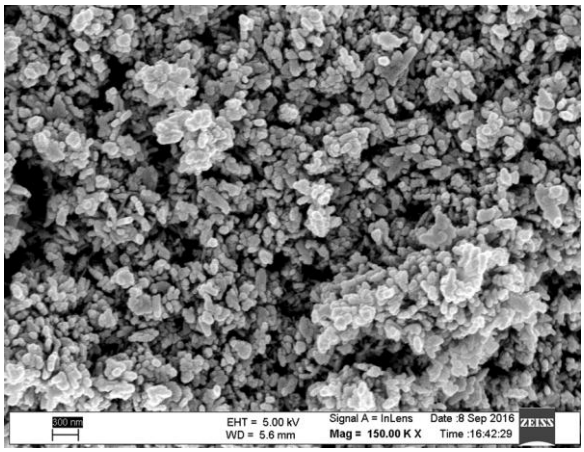


Fig 4. FTIR spectra of CuO nanoparticles prepared at 0.2M, 0.4M and 0.6M

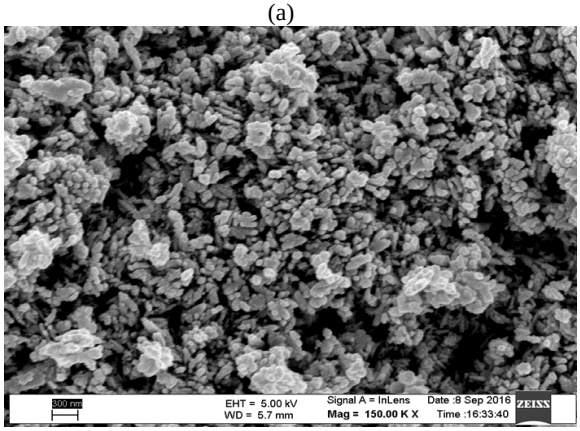
KBr pellets technique was used to find the FTIR spectra of prepared CuO nanoparticles. Fig.4 shows the FTIR spectra of CuO nanoparticles prepared at 0.2M,0.4M and 0.6M respectively in the range of 4000–400 cm^{-1}. Formation of highly pure CuO nanoparticles was confirmed by bands at around 605.55, 536.76cm^{-1} of FTIR spectra which are formed due to vibrations of Cu(II)-O bonds. The distinctive peaks of CuO are located in between 984 cm^{-1} to 426cm^{-1}. A wide absorption band at around 3480.91cm^{-1} is observed which occurs due to absorption of moistures by the nanoparticles. The metal-oxygen bond is observed at 1341.39 cm^{-1}, 1502.15cm^{-1} and 1651.72cm^{-1} indicating the formation of CuO from Copper Chloride. All the allocation of peaks is in accordance with the values found in the literature [26-32].

D. SEM Analysis

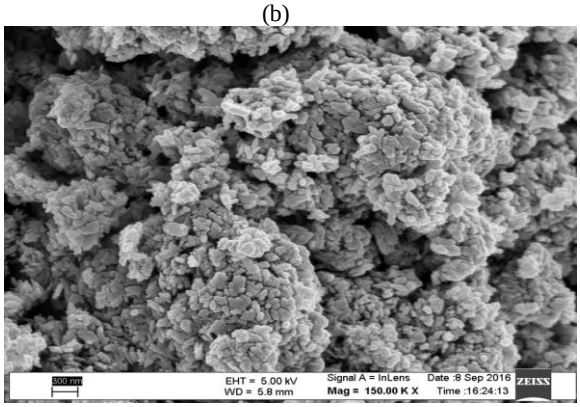
The surface morphology and micro structure of prepared CuO nanoparticles were investigated with the help of SEM images as shown in figure 5. It clearly shows the almost spherical morphology of prepared CuO nanoparticles with a homogeneous distribution. Agglomeration of spherical CuO nanoparticles was also observed from the SEM images.



(a)



(b)



(c)

Fig 5. SEM images of CuO nanoparticles prepared at (a)0.2M, (b) 0.4M and (c) 0.6M molar concentration of precursors

IV. Conclusion and Future Scope

Copper oxide nanoparticles are successfully prepared by sol-gel method. XRD pattern shows the prepared nanoparticles are of the monoclinic structure. It is observed that with the increase of crystallite size, the dislocation density decreases. With the increase in molarity of precursors, the band gap of prepared nanoparticles decreases. FTIR spectra show the stressing modes of the prepared samples. SEM images show the morphology of prepared CuO nanoparticles. We plan to study the prepared CuO nanoparticles as water purifier as our future work.

Acknowledgment

We express our sincere gratitude to SAIF, Dept. of Instrumentation & USIC, Gauhati University and IIT Guwahati for providing different instruments facility.

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