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RESEARCH ARTICLE

Flower-Shaped CuO Nanostructure Synthesized by Sonochemical Method and the effect of NaOH concentrations

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Abstract

Copper oxide nanoparticles were successfully fabricated using Sonochemical method. The synthesized CuO nanostructures were characterized by X-ray diffraction (XRD), UV-vis spectroscopy and Fourier transform infrared (FT-IR) spectroscopy. The effect of pH on the final product was investigated. CuO nanoparticles were synthesized by drop wise addition of Cu $(CH_3COO)_2$ solution to NaOH, in two different concentrations and pH maintained at 9. The study helps produce CuO nanoparticles easily at large scale. Besides, the method is simple to synthesis, easeful, flexible, fast, cost effective, and pollution free.

INTRODUCTION

Synthesis of nanostructured oxide materials paid significant attention in the last few years (Allahyari *et al.* , 2014,Chang *et al.* , 2010,Abulizi *et al.* , 2014). The metal oxides are extremely important technological materials for use in electronic and photonic devices and as catalysts in chemical industries. The metal oxides are also often used as active materials of dental cements. In recent years, copper-based nanomaterials have received astonishingly great attention in the applications of optoelectronic devices, catalysis and superconductors. CuO is a p-type semiconductor (Eg = 1.2eV) with excellent photovoltaic, electrochemical, and catalytic properties. Also, it is inexpensive, non-toxic, and readily available. Copper oxide nanoparticles are used in a wide range of applications such as gas sensors, magnetic storage media, solar energy transformation, semiconductors and organic catalysis (Anandan *et al.* , 2007,Kim *et al.* , 2008). For synthesizing copper oxide nanoparticles, many efficient approaches have been carried out such as sol–gel technique (Suleiman *et al.* , 2013), alkoxide based preparation, microwave irradiation, Precipitation-Pyrolysis and thermal decomposition (Zhang *et al.* , 2006,Sabbaghi *et al.* , 2012).

In addition, most of the pathways suggested for the synthesis of CuO nanoparticles involve environmental malignant chemicals and organic solvents which are toxic and not easily degraded in the environment (Volanti *et al.* , 2008). Environmental friendly synthesis requires alternative methods such as ultrasound, hydrothermal, green synthesis and laser. Sonochemical method is particularly attractive because it is inexpensive, safe, environmentally benign and bestowed with many virtues especially under supercritical conditions.

We decided to apply the Sonochemical approach to copper metal. Interestingly, uniform sized nanoparticles were obtained by a simple reaction of copper powder. The reported method is economical, fast, environmentally benign and free of pollution, which makes it suitable for large scale production (Tiwari *et al.* , 2008,Sun *et al.* , 2005). The aim of the study is to provide feasibility of the simple route for the preparation of copper oxide nanostructures without additives. The prospects in the process are bright and promising (LIU *et al.* , 2012,Suleiman *et al.* , 2013). Using ultrasound to produce nanomaterials is a developing and promising area of scientific researches. Applying

ultrasonic radiation offers significant advantages in many cases and sometimes it is the only effective solution for problems related to synthesis and further application of nanoparticles(Karthik *et al.* , 2013,Suramwar *et al.* , 2012,Lanje *et al.* , 2010).

In this paper, we have synthesized CuO nanoparticles by the simple Sonochemical method at a nanosize. The synthesized nanoparticles were characterized by XRD, SEM, UV and FTIR spectrometer (Padil *et al.* , 2013).

Material and Methods

2.1 Materials

All chemicals used in the experiment were analytic reagent grade. Copper (II) acetate $Cu(CH_3COO)_2$, and Sodium Hydroxide (NaOH) were purchased from Sigma-Aldrich, Germany. Ethanol and Acetone were purchased from Merck Chemicals (India) Pvt. Ltd. Double Deionized water was used throughout the experiment.

2.2 Synthesis of CuO nanoparticles

Sonochemical method was used for the synthesis of CuO nanoparticles. The reaction was based on preparing 25 ml of 0.3 M aqueous solution of NaOH by mixing it slowly in 60 ml of 0.5 M aqueous solution of copper acetate dehydrate under continuous stirring for 60 min. By following the same process, instead of 25 ml of 0.3 M aqueous solution of NaOH, 25 ml of 0.5 M aqueous solution of NaOH was used and results were analyzed. The Sonication process of the resultant solution took 4 hours at room temperature and pH was maintained at 9. The precipitate was in black color and filtered using Whatman filter paper.

Further, distilled water, acetone and ethanol were used several times to wash the precipitate and dried at 300°C for 3 h in hot air oven. Finally, the dried material was ground using Agate Mortar. The concentration of NaOH played a vital role in the formation of CuO nanoparticles since OH- was strongly related to the reaction that produces nanostructures.

2.3 Characterization

2.3.1 UV-vis and FTIR techniques

UV-Vis spectra were obtained using Perkin Elmer UV-Vis Spectrophotometer. IR absorption spectra were recorded in a FT-IR Spectrum 1000 Perkin Elmer spectrometer on thoroughly dried samples using KBr as dilutant (Rahimi *et al.* , 2010).

2.3.2 X-ray diffraction analysis

The crystal structures of the samples were examined by X-ray diffraction, XRD (D8 Advance Bruker Co.,) at CuKα (λ = 1.54056 A) in the 2θ range from 5° to 80° in steps of 0.03° with a count time of 0.4s (Zhu *et al.* , 2004,Al-Mosawi *et al.* , 2015). The crystallite sizes of the particles were calculated by using Scherrer's equation.

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

Where,
D is the crystallite size of the particles,
K is a shape factor (K=0.9 in this work),
λ is the wavelength of the incident X-ray (1.54056 A, CuKα),
θ is the diffraction angle and β is the fullwidth half maximum.

2.3.3 Scanning electron microscopy

The morphologies of the products were examined using a Quanta-200 scanning electron microscope (El-Meligy *et al.* , 2015,Fouad, 2014). Samples were gold-coated prior to scanning electron microscopy (SEM) analysis.

3. Result and Discussions

3.1 UV-Visible absorption analysis of CuO NPs

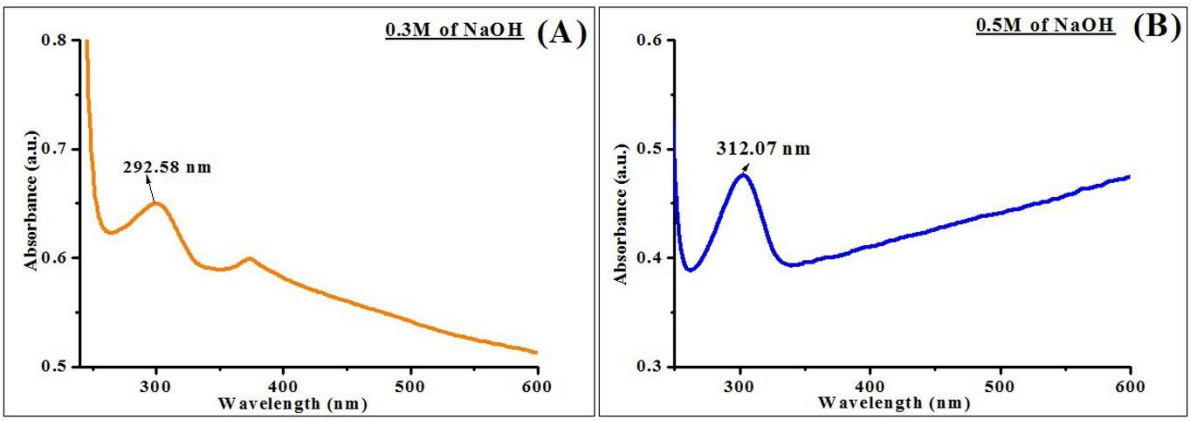

Fig. 1. Shows the absorption spectra of CuO nanoparticles synthesized at two different concentrations of NaOH at (a) 0.3M and (b) 0.5M

UV-Vis spectral analysis was carried out between the ranges of wavelength 200 to 1600 nm. This behavior can occur for a variety of reasons (Li *et al.* , 2004), such as internal electric fields within the crystal, deformation of lattice due to strain caused by imperfection and inelastic scattering of charge carriers by phonons. UV- vis spectrum of CuO nanoparticles (0.3M of NaOH and 0.5M of NaOH) and their broad absorption peaks are observed at 312.07 and 292.58nm respectively, which can be attributed to the quantum confinement effect.

3.2 FTIR of CuO NPs

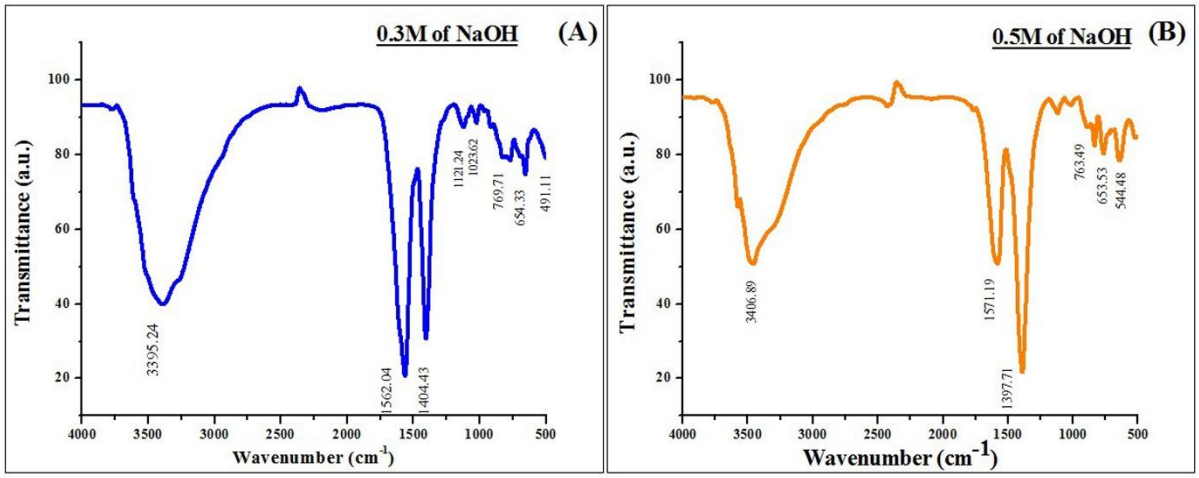

Fig. 2. FTIR spectra of CuO nanoparticles synthesized at two different concentrations of NaOH at (a) 0.3M and (b) 0.5M

The composition and quality of the CuO nano particles were analyzed by FTIR, in the range of 400-4000 cm^{-1}. The absorption bands at 491.11, 654.33, 769.71and 1023.62 cm^{-1} were observed from the FTIR spectrum which is due to the CuO stretching (Anandan *et al.* , 2012). Weak and broad absorption bands at 1562 and 3395cm^{-1} were also absorbed due to the existence of water molecules at 1404 cm^{-1} and C-H stretching vibrations (Deng *et al.* , 2011).

3.3 XRD of CuO NPs

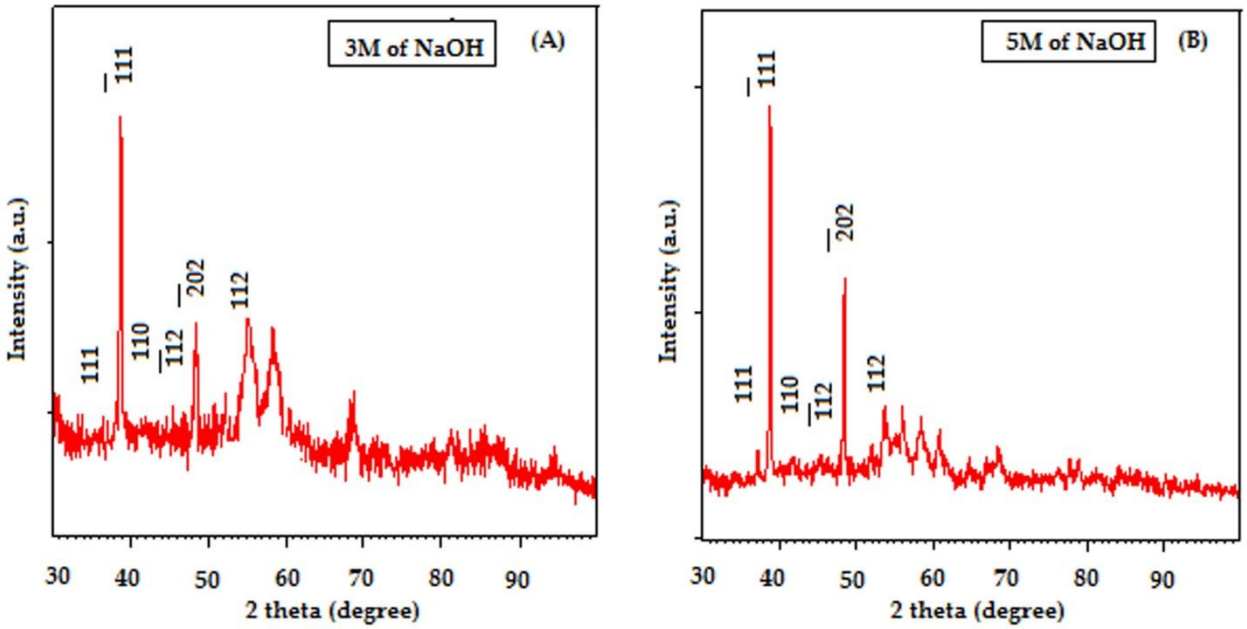

Fig. 3. Shows XRD spectra of CuO nanoparticles synthesized with two different concentrations of NaOH at (a) 0.3M and (b) 0.5M.

The values of the lattice parameter were determined from the X-ray diffractogram using powder X software, which clearly matches with the reported values of lattice parameter of CuO. The presence of a relatively sharp peak in the background of a wider peak suggests that both large and small grains are collected together (Guo *et al.* , 2012,Meshram *et al.* , 2012). The Scherrer's formula was used for calculating the nanoparticle size of the sample (powder). It shows the monoclinic structure and the positions of peaks are in good agreement with the reported values given JCPDS NO. (48-1548).

From the graph (A) the major peaks located at 2θ = 18.780 and 28.4130. The calculated average grain size was around 35.88nm and graph (B) the major peaks were located at 2θ = 18.80 and 38.50. Finally, the calculated average grain size was found to be 19.93nm.

3.4 SEM images of CuO NPs

The SEM image of bare CuO nano particles indicated that the CuO nanoparticles have flower like shape and favorable property exhibiting better photo catalytic activity and agreed well with average crystallite size (Vaseem *et al.* , 2008a,Vaseem *et al.* , 2008b). In the case of bare and surface modified CuO nanoparticles significant effects were found over the size control and had no agglomeration.

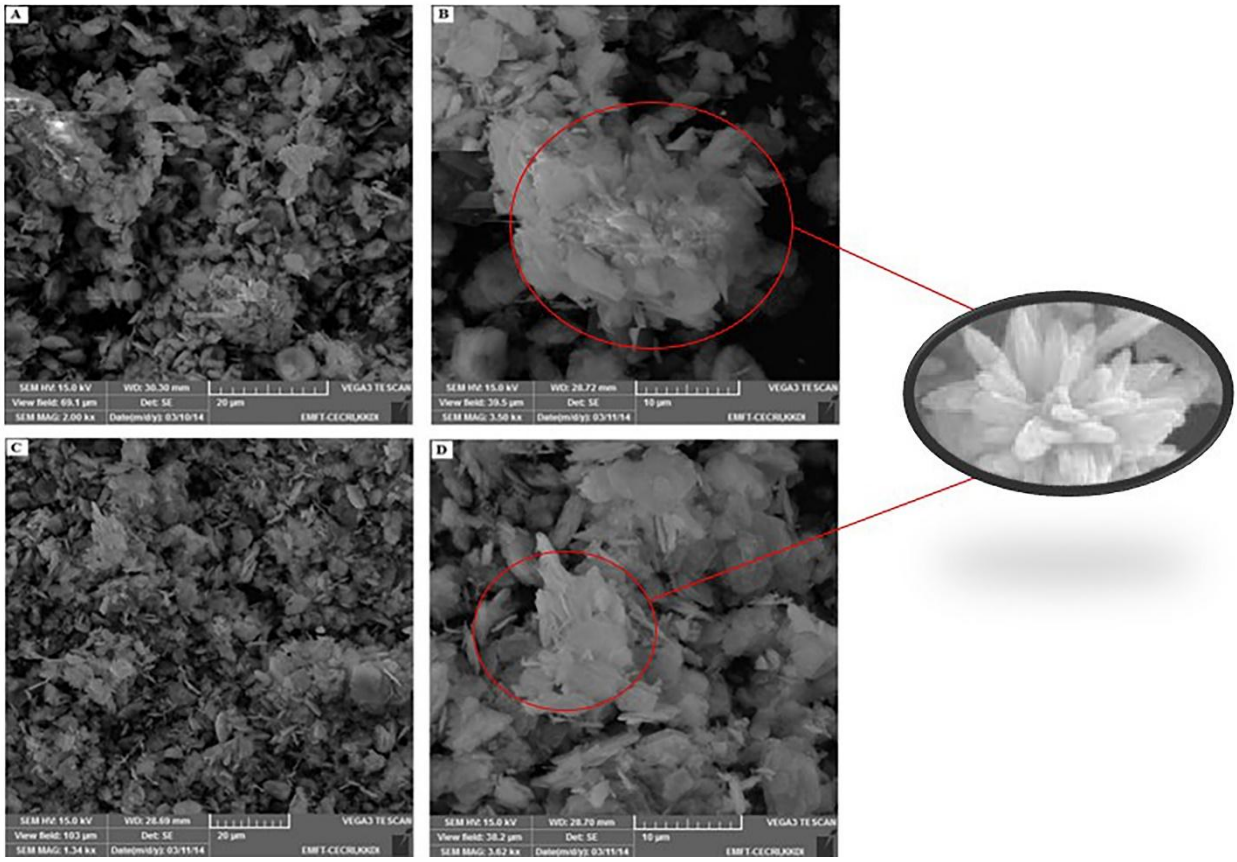

Fig. 4. Shows SEM image of CuO nanoparticles synthesized with two different concentrations of NaOH at (a & b) 0.3M and (c & d) 0.5M

4. Conclusion

The CuO nanoparticles were successfully synthesized by Sonication method. The composition and quality of the nanoparticles were analyzed by FTIR studies. The XRD confirms the crystal structure and phase purity of the sample. SEM images indicate that CuO nano particles have flower like shape. The surface morphology of CuO was studied by SEM technique that shows that the morphology of the nanoparticles is strongly dependent on NaOH concentration. The grain size of the particles is calculated from the Scherrer's formula by using XRD results. The study shows that 0.3M of NaOH results in average grain size around 35.88nm and 0.5M of NaOH results in average grain size around 19.93nm. When the concentration of NaOH increases the size of nanoparticles also decreases.

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