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Effect of CuO Nanoparticles on Antimicrobial Activity Prepared by Sol-Gel Method

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Abstract

Study of structural and antimicrobial properties of copper oxide nanoparticles synthesized by sol-gel combustion method. XRD, FTIR techniques were used to characterize. The nanoparticles XRD pattern showed the crystalline of nature of CuO NPS. XRD spectra confirmed the formation of single phase CuO nanoparticles. Crystallite size was found to increase with an increase in annealing temperature due to atomic diffusion a minimum crystallite size of (15.3)nm was observed in the case of CuO NPS annealed at (200)◦C. Transmission electron microscopy results corroborate well with XRD results. CuO nanoparticles showed excellent antibacterial activity against various bacterial strains (E.Coli, P. aeruginosa) bacteria. The antibacterial activity of CuO NPS was found to be size dependent.

Keywords: CuO nanoparticles ,X-ray diffraction ,FTIR, antimicrobial activity.

INTRODUCTION

Metal oxide nanoparticles (NPs) have been receiving considerable attention for their potential application in photo electronics , Nano devices ,Nano electronics ,Nano sensors, magnetic storage media , solar energy ,and superconductors [1,2] .Among various metal oxide NPS ,CuO has attracted particular attention because it is the simplest member of the family of copper compounds and shows a range of useful physical properties such as high temperature superconductivity , electron correlation effect[3,4]. Some method for the preparation of Nano crystalline CuO have been reported such as the sono chemical method [5] , sol-gel technique [6] one-step solid state reaction method at room temperature [7] , electrochemical method [8] , and thermal decomposition of pre cursors [9] , copper oxide used as an antimicrobial gent, and CuO nanoparticles have been investigated previously for enhancing antibacterial properties [10,11] . To realize the potential of CuO nanoparticles to act as antimicrobial agent, we synthesized different sized CuO nanoparticles, by controlling the annealing temp during the sol-combustion synthesis. Furthermore, the bacterial activities of CuO nanoparticles against to E.Coli, P. aeruginosa bacteria were investigated.

EXPERIMENTAL WORK

Synthesis of CuO nanoparticles

CuO NPs were synthesized by sol-gel method. Using [$Cu(NO_3)_2.3H_2O$] and citric acid dissolved in di-ionized water with amorality (0.1M) .the solution was stirred a magnetic stirrer at 90◦c . Stirring continued until gel-formation. After wards, the gel was allowed to burn at 100◦c .Which was further annealed for 2 hours at varying temperature 200,300,400,500,600◦c**,**to obtain the highly crystalline CuO nanoparticles.

Characterization of CuO nanoparticles

The crystalline nature of CuO was carried by XRD. Crystallinity structure and crystallite size of CuO nanoparticles were determined by XRD technique using (XRD-6000, SHIMADZU) with cu-kα radiations (λ=0.15406 nm) in 2ϴ rang from 20° to 80°. FTIR spectra of the samples were obtained using (IRAffinitiy -1, SHIMADZU) FTIR spectra-photometer.

Antibacterial activity of CuO NPs.

Antibacterial activities were determined by the well –diffusion method. Antibacterial activities of the synthesized CuO nanoparticles of different sizes were determined using E.Coli and P. aeruginosa bacteria. Were cultured in nutrient broth at 37◦c [12] .Bacterial sensitivity to antibiotics or nanoparticles is commonly tested using a well diffusion assay , utilizing antibiotics or nanoparticles impregated disks[13] prepared CuO nanoparticles suspensions was added into the wells bacteria culture was prepared by spreading 100μL culture bron having 10^6 cfv/ml of each test organism on . solid mullerhinton a gar plates . The plates were allowed to stand for 15 minutes to allow for culture absorption . The (6 mm) size wells punched into the agar with the head of sterile micropipette tips. Wells were sealed with one drop of molten agar (0.8٪) Muller hinton agar to prevent leakage from the bottom of the plate using a micropipette , 100 μL (50 μg) of the nanoparticles solution sample was poured in to each of six well on all plates after incubation at 37◦c for 24 hours . The size of zone of inhibition was measured.

THE PROCEDURE AND THE RESULT

Structural Properties CuO nanoparticles.

Fig (1) shows the XRD pattern of CuO NPS . All the peaks of CuO nanoparticles can be indexed to the monoclinic crystal system CuO (JCPDs card no. 05-0661). No characterizes peaks of anyimputties was observed in XRD pattern , showing single phase sample for material ,suggested that high quality of CuO nanoparticles was prepared.

The crystallite size was calculated using scherrer equation [14]

$$G(x) = \frac{1}{\delta\sqrt{2\pi}} exp\frac{(x-Q^2)}{2\delta^2} \ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

where k=0.9 is the shape factor, λ is the wave length of x-ray radiation , β is the full width at high maximum (FWHM) of the peaks at the diffracting angle Θ . Crystallite size calculated by the sherrer equation was found to be (15.3)nm. The XRD spectra of CuO nanoparticles annealed at different temp. It is clear that the intensity of crystalline peaks increase with temp,indicating an improvement in the samples crystallinity . Simultaneously , the peaks become narrower as the temperature increase resulting in the increase of crystallite size. The variation of crystallite size with temperature was calculated are represented in figure(2). It can be seen from figure(2) that crystallite size increase with the increase annealing temp. The increase in crystallite size with temperature can be attributed to atomic diffusion.

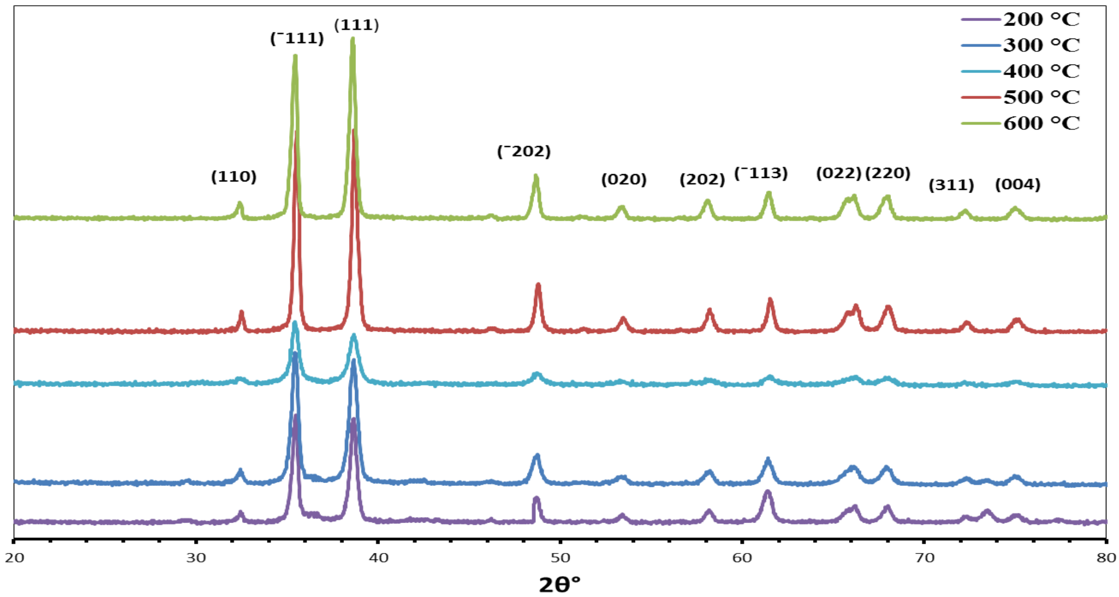


Figure 1. Shows the XRD pattern of CuO NPS at different temperature

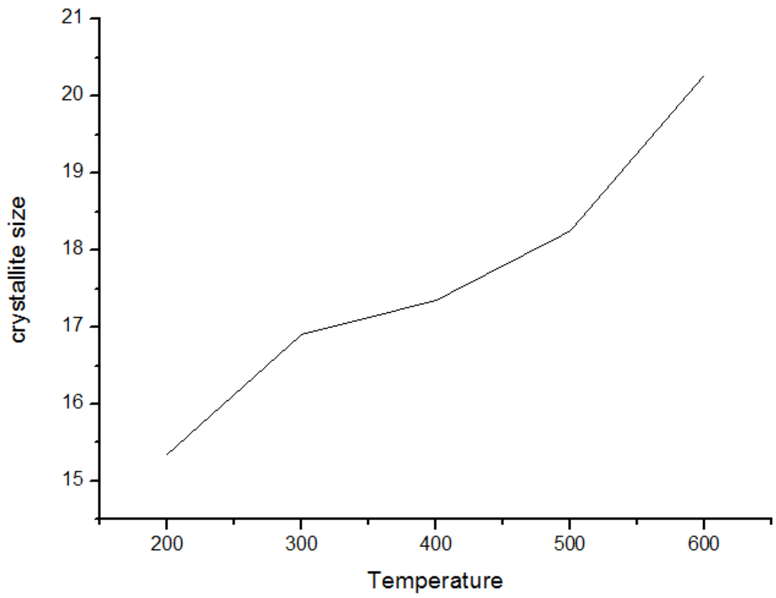


Figure 2. The variation of crystallite size with temperature

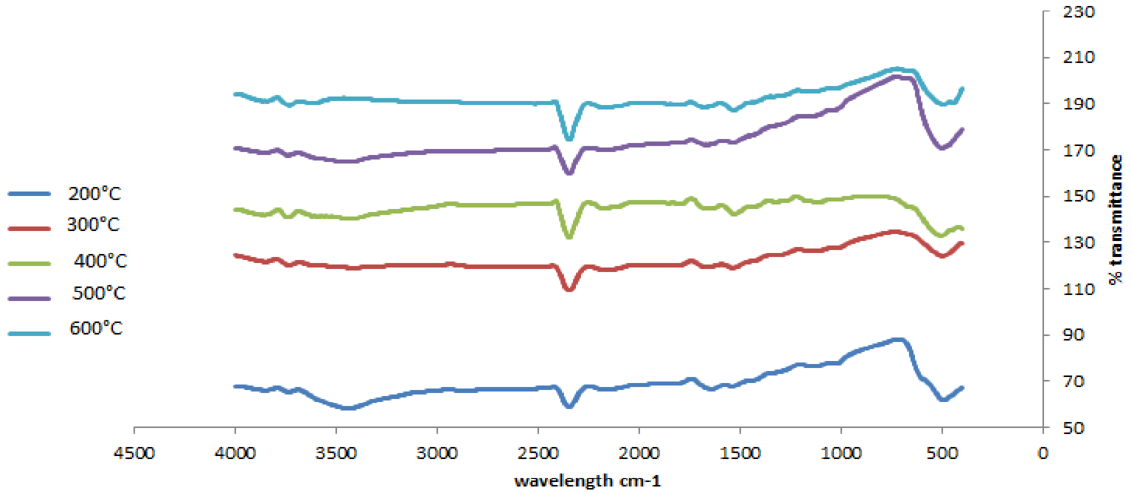
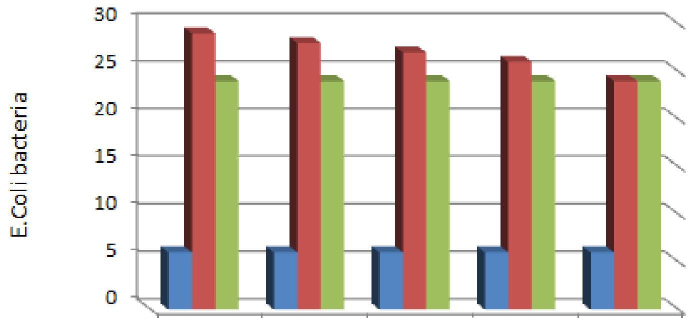
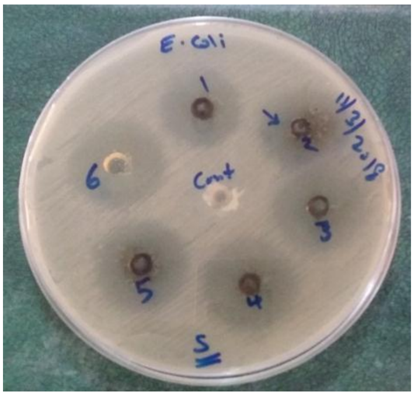


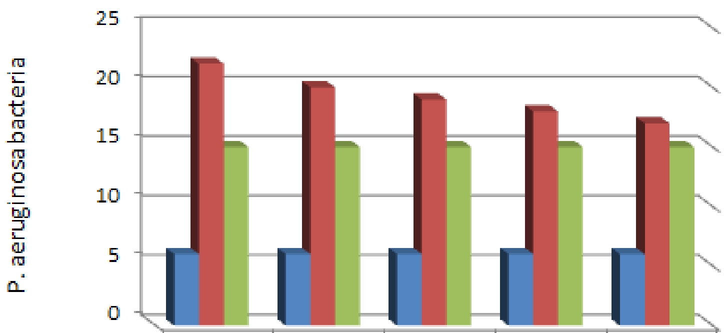
Figure 3. FTIR spectra of CuO NPs annealed at different temperature.



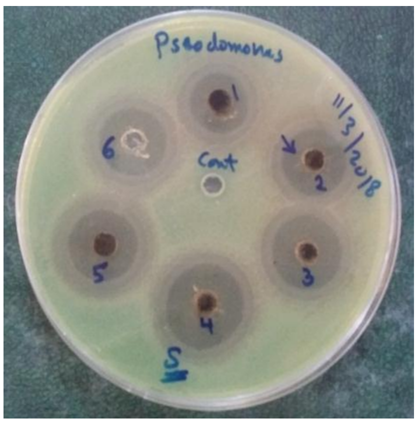
Temp	200◦C	300◦C	400◦C	500◦C	600◦C
Negative cont	6	6	6	6	6
Zone inhibition	29	28	27	26	24
Antibiotic	24	24	24	24	24



(4a)



Temp	200◦C	300◦C	400◦C	500◦C	600◦C
Negative cont	6	6	6	6	6
Zone inhibition	22	20	19	18	17
Antibiotic	15	15	15	15	15



(4b)

Figure 4. Histogram representing the zone of inhibition of CuO NPs and Antibiotic against (a) E. Coli bacteria, (b) Paeruginosa, numbers (1,2,3,4,5) represented the concentration at temperatures (200,300,400,500,600) ◦C, and number (6) represented the tetracycline antibiotic.

FTIR

Spectra were recorded in solid phase using the KBr pellets technique in the rang of 4000-400 cm^{-1}. FTIR spectra of CuO nanoparticles treated at 200◦C , 300◦c,400◦c,500◦c,600◦c are shown in fig (3) .FTIR spectra exhibiting only three variation : occurring at approximately 500 cm^{-1} for all samples ,which can be attributed to the vibrations of Cu-O , conforming the formation of highly pure CuO nanoparticles . A weak band at round 2340 cm^{-1} may be attributed to the vibration of atmospheric CO_2 . These assignments are in agreement with the values available in literature [15-17].

Antibacterial activity of CuO nanoparticles

Antibacterial activity of CuO NPs was analyzed against various bacterial strain (E.Coli, P. aeruginosa) .Fig (4) represent the antibacterial activity of CuO NPs for various bacteria in a well diffusion assay . Results showed that CuO NPs demonstrated excellent antibacterial activity against a rang of bacteria .The diameter of inhibition zone reflects magnitude of susceptibility of microbes .The smallest CuO nanoparticles (particle size 15.3 nm) synthesized at the lowest temperature of 200◦C.

SUMMARY AND CONCLUSION

Highly pure CuO nanoparticles were prepared by using a sol-gel combustion method. XRD spectrum revealed that CuO nanoparticles were monoclinic. XRD spectra confirmed the formation of single phase CuO nanoparticles. Crystallite size was found to increase with the increase in annealing temperature. Minimum crystallite size of (15.3) nm was observed in the case of CuO nanoparticles annealed at 200◦c .FTIR also validated the purity of CuO nanoparticles . CuO nanoparticles showed excellent inhibition for all the microorganisms reached a maximum point using CuO nanoparticles annealed at 200◦c.

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