CuO:Fe nanoparticles: preparation, characterization and antibacterial properties

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Abstract:

$Cu_{1-x}Fe_xO$ (x=0,0.05,0.10,0.15) nanoparticles with average diameter of about 37 nm were prepared by sol–gel method at 400°C. The obtained nanoparticles were characterized by X-Ray Diffraction (XRD), Transmission Electron Microscopy (TEM), an Ultraviolet–Visable (UV–vis) spectroscopy. Optical studies show that Optical band gap of products increase with doping. X-ray diffraction patterns of samples confirm the presence of cupric oxide (CuO) phase. The antimicrobial properties of CuO:Fe nanoparticles with different Fe concentration were investigated using Escherichia coli (E.coli) bacteria.

KEYWORDS : *CuO nanoparticle, Antibacterial properties, sol-gel method*

INTRODUCTION

Copper oxide-based materials have been widely investigated due to their potential applications in many fields. Two common forms of Copper oxide are cuprous oxide or cuprite (Cu_2O) and cupric oxide or tenorite (CuO) [1]. Cupric oxide (CuO, *tenorite*) is a monoclinic p-type semiconductor with a narrow band gap of 1.2–1.5 eV at room temperature with lattice parameter a =4.6837A° , b=3.4226 A° , c=5.1288 A° and β=99.54 ° [2], whereas cuprous oxide (Cu_2O, *cuprite*) is a cubic (a=4.253 Å) p-type semiconductor with a direct band gap of 2.0 eV [2,3].

Transition metal oxide such as copper oxide (CuO and Cu_2O), iron oxide (FeO, Fe_2O_3 or Fe_3O_4) and zinc oxide (ZnO) nanomaterials have special physicochemical properties arising from the quantum size effect and high specific surface area, which may be different from their atomic or bulk counterparts [4]. CuO nanoparticles (NPs) suspension (nanofluid) has excellent thermal conductivity for it to be used as a heat transfer fluid in machine tools[5]. The bactericidal effect of metal NPs has been attributed to their small size and high surface to volume ratio, which lead to closer interaction with microbial membrane [6].

Till now there has not been any report on studing the effect of Fe_2O_3 doping as antibacterial. In this work nanoparticles of CuO with different Fe concentration were successfully synthesized via sol-gel method, and its antibacterial activity, in addition to structural and optical properties were studied.

EXPERIMENTAL

CuO:Fe nanoparticles were synthesized by sol–gel method. A precursor solution was prepared by use of ethanol (C_2H_5OH, Merck, >99.9%) and deionized (DI) water as solvent (1:1). Then, copper nitrate [$Cu(NO_3)_2.3H_2O$] and iron nitrate [$Fe(No_3)_3.6H_2O$] were added. Citric acid and ethylene glycol used as polymerization and complexing agents, respectively. After 1h of stirring at 40 ºC a green solution was obtained. The homogeneous mixture was maintained under reflux at 100-110°C for 4 hours. After vaporizing the excess solvents, a wet gel was attained. Finaly, the black powder was calcined at 600°C for 1 h in oven and then milled.

The samples were characterized with using X ray diffractometer (D8 Adnance Bruker) (Cu K_α radiation of wavelength λ=1.5406 Å). The intensity was determinded in the range 20º< 2θ< 80º with a scanning step size of 0.04º. The average crystallite size of powders was estimated using the Scherrer's relation.

Transmission electron microscope (TEM) (LEO 912 AB) was also used for estimation of crystalline structure, morphology and mean size of NPs.

To make nanoparticle CuO suspension (nanofluid) for antibacterial test a preset amount of NPs was mixed with distilled water ,at the concentration of 1g/l, with vigorous stirring in an ultrasonic bath. for more homogenous nanofluid, a ball-mill mixer (Retsch MM400, 28 Hz) for 5 min was used. Nanofluids prepared were autoclaved at 121° C for 15 min .

Antibacterial test was done by measuring growth curve of gram-negative *E . coli* (HB 101) incubated in the LB broth medium in presence of nanofluids. The growth curve was determined by measuring time evaluation of optical density (OD) of the samples. The measurements were done at 600 nm with spectrophotometer (WPA LightWave S2000 UV/Vis spectrophotometer) at the frequency of once an hour.

RESULTS AND DISCUSSION

Fig 1. displays the XRD spectra of the nanoparticles heated at 400 ºC. According to the literatures [7] two reflection at 2θ =35.6 [002] and 2θ=38.8 [111] were observed in the diffraction patterns and were ascribed to the formation of the CuO (space group C2/c) monoclinic crystal phase. As can be seen, the [400] peak related to cubic γ - Fe_2O_3, disappear in samples with 15% amount of Fe and it can described that Fe^{+3} can be incorporated into the CuO lattice with no phase segregation taking place because Fe atom has rather a similar ionic radii with Cu atom (0.73 A° for Cu and 0.645 A° for Fe).

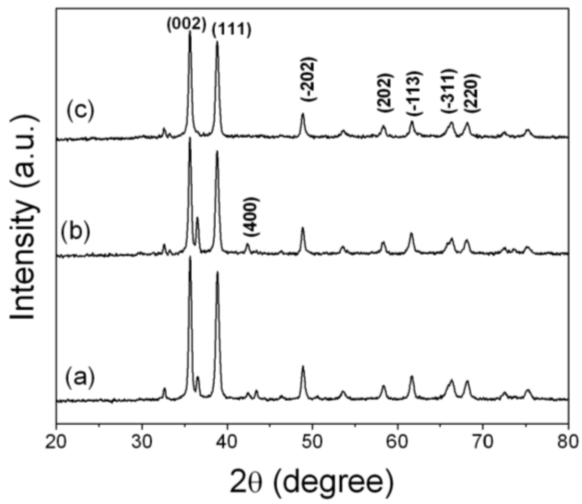

Fig 1.XRD Pattern of CuO:Fe nanoparticles with a)5% b)10% and c)15% of Fe content.

Fig 2. shows a typical TEM micrograph of CuO:Fe nanoparticles calcined at 400ºC. An agglomeration of nanoscale particles is clearly visible, showing a uniform distribution of particle sizes and a homogeneous morphology. Particle-size distribution histogram of CuO:Fe nanoparticles (shown as the bottom inset of Fig 2.) indicated that average diameter of nanoparticles counted from TEM image are about 37 nm.

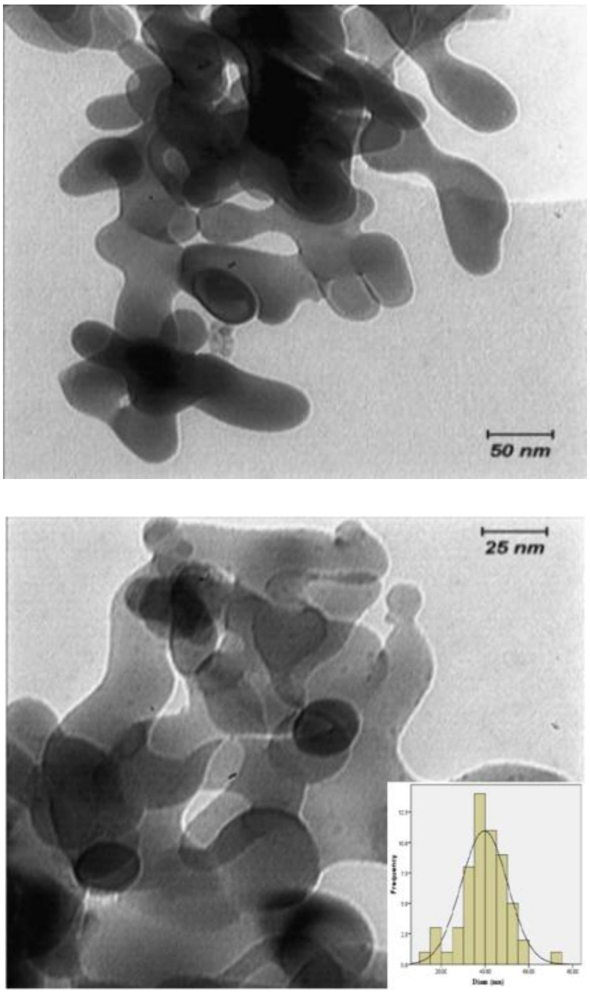

Fig 2. TEM images of CuO:Fe NPs with 10% of Fe content and Particle-size distribution histogram.

Figure 3. shows growth curves of bacteria in the present nanoparticles with the negative control and shows the effect of doping on antibacterial activity of NPs. Recent studies have shown that copper alloy surfaces kill *E. coli* [8,9]. As it can be clearly seen by increase in doping level, the antibacterial activity slightly increase. The antibacterial mechanism of copper NPs has been attributed to the fact that Cu^{2+} ions eluted from NPs are absorbed by bacteria when the NPs concentration is high enough [10] and they are also small enough to disrupt bacterial cell membranes and gain entry in order to disrupt enzyme function [11].

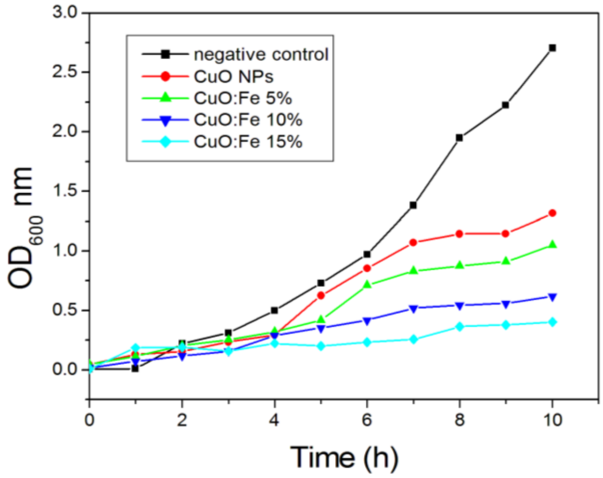

Fig 3. growth curve of *E.coli* in LB medium in the presence of CuO NPs with different dopant concentrations

CONCLUSIONS

CuO:Fe nanoparticles with sizes ranging from 10 to 60 nm (based on TEM) were synthesized successfully by sol-gel method at 400ºC. The effects of addition of impurity on the physical properties of the NPs have been investigated. XRD patterns show the formation of the CuO monoclinic crystal phase. The histogram of particle size shows that size of majority of the particles is in the range of 30–50 nm. The CuO:Fe NPs with 15% of Fe content exhibited a strong antibacterial activity against *E. coli* bacteria.

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