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Synthesis, characterization and evaluation of antibacterial activity of PEG-CuO nanoparticles

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Abstract : Copper oxide nanoparticles (PEG-CuONPs) were synthesized by an aqueous precipitation method using Copper acetate as a precursor, polyethylene glycol (PEG) which acts as a capping agent and KOH as a stabilizing agent. This is a simple and cost effective method which gives a large scale production of CuONPs. A detailed characterization of the synthesized nanoparticles was performed by using UV-Visible spectroscopy (UV-Vis), Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) studies,and Scanning electron microscopy-Energy dispersive spectroscopy (SEM-EDS). The average crystallite size of CuONPswas determined by Debye-Scherer formula it was found to be 48 nm. The investigation of antibaterial activity of PEG-CuONPs was tested against*Bacillus Substillis, Klebsiella pneumonia, Pseudomonas putida* which was proved to be effective results.

Keywords : PEG-CuONPs, polyethyleneglycol, aqueous precipitation method, antibacterial activity.

Introduction

Nanoparticles(NPs) are small particlesbetween 1to100 nanometres in size. There is a great difference in physical and electronic properties of the nanometre scale particles compared to bulk materials.The transition from bulk to nanoparticles leads to the display of quantum mechanical properties and an increased dominance of surface atoms which increases the chemical reactivity of a material. A notable example includes the tunable band gap[1] and catalytic behaviour of nanoparticles[2]. The nanoparticles synthesized by solution based chemical methods, a capping agent which adsorbs to the nanoparticles surface, generally is added to control the size of nanoparticles as well as prevent the agglomeration of the particles. Polymers are chosen as good host materials because they usually exhibit long term stability and possess flexible reprocess ability. In addition, transition metal oxide nanoparticles are an important class of semiconductors, which have applications in magnetic storage media, solar energy transformation, electronics and catalysis[3-11]. Among the transition metal oxide nanoparticles, Copper oxide nanoparticles are of special interest because of their efficiency as nanofluids in heat transfer application, for example, it has been reported that 4 % addition of CuO improves the thermal conductivity of water by 20 %[12]. CuO is a semiconducting compound with a narrow band gap and used for photoconductive and photothermal applications[13]. However, the reports on the preparation and characterization of nanocrystallineCuO are relatively few to some other transition metal oxides such as Zinc oxide, Titanium dioxide, Tin dioxide and Iron oxide. Some methods for the preparation of nanocrystallineCuO have been reported recently such as the sonochemicalmethod[14], sol–gel technique[15], and one-step solid state reaction method at room temperature[16], electrochemical method[17], thermal decomposition[18] and co-implantation of metal and oxygen ions[19] and so on.The use of CuO nanoparticles in nanoformulations relies on their antimicrobial

ability that allows for the development of multiple products, from antimicrobial solutions utilized to disinfect the surfaces and medical devices to antimicrobial wound dressings, textiles and coatings. In view of this, we have synthesized Copper oxide nanoparticles with controlled size in liquid medium by simple aqueous precipitation method containing PEG (polyethylene glycol) as a capping agent.Polyethylene glycol (PEG) is a cheap non-ionic surfactant that is used for the synthesis of metaloxides[20]. It is also used in many biomedical applications, especially drug delivery since it offersa good biocompatibility to the whole structure which contains it[21]. PEG is the most commonlyutilized variant due to its lower toxic[22].We reported heresynthesis, characterized CuONPsby employing various chemo-physical techniques and investigated their antibacterial activity.

Experimental

Materials

All chemicals used in the experiment are analytic reagent grade. Copper acetate monohydrate $Cu(CH_3COO)_{2.2}H_2O$ and Potassium hydroxide (KOH) pellets were purchased from Merck, India. Polyethylene glycol (PEG)was purchased from LobhaChemie, India.The bacterial test strains were procured from IMTECH, Chandigarh, India. The chemicals used in antibacterial studies obtained from Himedia laboratories, India.

Methods

The absorption spectra of synthesized PEG-CuONPswere recorded on UV-Visible spectrometer UV-3600 series, Shimadzu in the range of 200-800nm. A small amount of PEG-CuONPs were dissolved in distilled water and spectra were recorded. The FTIR spectra of the samples were recorded with Shimadzu spectrometer in the range of 4000-400 cm^{-1} using KBR pellets technique. The powder X-ray diffraction (XRD) of the sample was performed using Philips Holland, XRD system PW 1710 with nickel-filtered CuKα (λ=1.5405 Å) radiation. The average crystallite size has been calculated from the line broadening using Scherer's relation. The SEM images of the synthesized PEG-CuONPswere carried out by using model ZIESS Special Edition 18. A Thin film of the sample was prepared by dropping a small amount of the sample on the carbon coated carbon grid, then the film on the SEM grid was allowed to dry under the mercury lamp for 5 minutes and the images were taken. The energy dispersive spectroscopy microanalysis system which automatically identifies the elements corresponded to the peaks in the energy distribution. For evaluation of antibacterial activity,the well-known techniqueis well diffusion method[23]was chosen to perform this test.All sterilized labware was used to study the activity. Fresh cultures of three different bacteria such as *Bacillus subtillis, Pseudomonas putida, Klebsiella pneumonia* was prepared and investigated against6 different concentrations of synthesized PEG-CuONPswhere ampicillin drug used as a control sample. After inoculation of all concentrations of samples, plates were incubated at 37^oC for 12 hours.

Synthesis of CuONanoparticles

An aqueous solution of Copper acetate (0.2M) was prepared in clean round bottom flask. Then PEG was added to the above aqueous solution and heated to 100^oC with constant stirring and 0.1M KOH solution was added to above heated solution till pH reaches to 7. The color of the solution turned from light blue to black and reaction was continued for another 2 hours. A large amount of black precipitate was formed. The precipitate was centrifuged and washed 3-4 times with deionized water. The obtained precipitate was dried in a hot-air oven for 24 hours and then obtained material was ground using mortar to form a fine powder which was further used for the characterization and anti bacterial activity.

Results and Discussion

UV-visible measurements

UV-visible spectrumis a very much useful technique in identification of nanoparticles. The absorption spectra of synthesizedCuO nanoparticles with capping agent PEG were shown in **Fig.1**.The synthesized PEG-CuONPs shows a peakin UV region at 370 nm indicating the formation CuO nanoparticles. The peak around the 370 nm was due to the surface Plasmon resonance (SPR) of metal oxide nanoparticles. The SPR was arisen due to the combined oscillationof the free conduction band electrons which is excitedby the incident

electromagnetic radiation. This type ofresonance is seen when the wavelength of the incidentlight far exceeds the particle diameter.

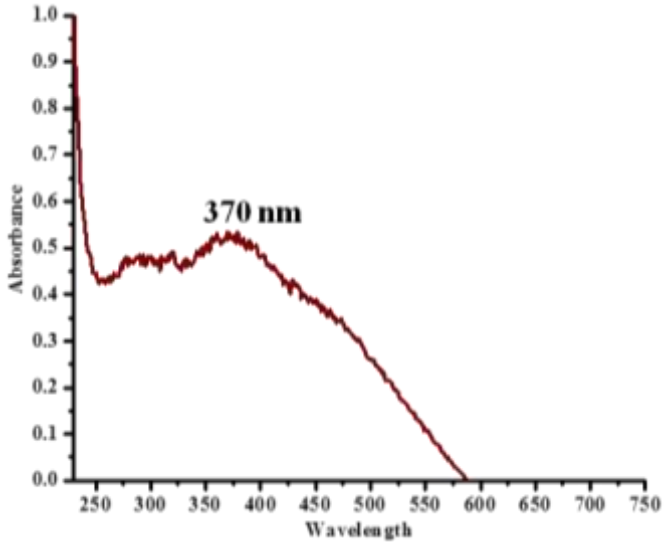

Fig. 1 Uv-visible absorption spectra of PEG-CuONPs

Fourier Transform Infrared measurements

The FTIR spectra of PEG-CuONPswere recorded in the range of 4000-400 cm-1 and are shown in **Fig. 2**. The peaks at 1640 cm^{-1}indicating the formation of coordinate bond between the nitrogen atom of the PEG and the copper ions, 1395 cm^{-1}indicates C-H scissoring and bending vibrations of alkanes, 1110 cm^{-1} due to -C-O-C stretching of ether. The peak at 871 $cm^{-1}$is attributed due to C−H out of planedeformation. The peak observed at 634 cm^{-1} in the spectrum of PEG-CuONPs is the characteristic of the Cu-O bond formation.This FTIR spectrum confirms the formation of CuONPs capped with the PEG.

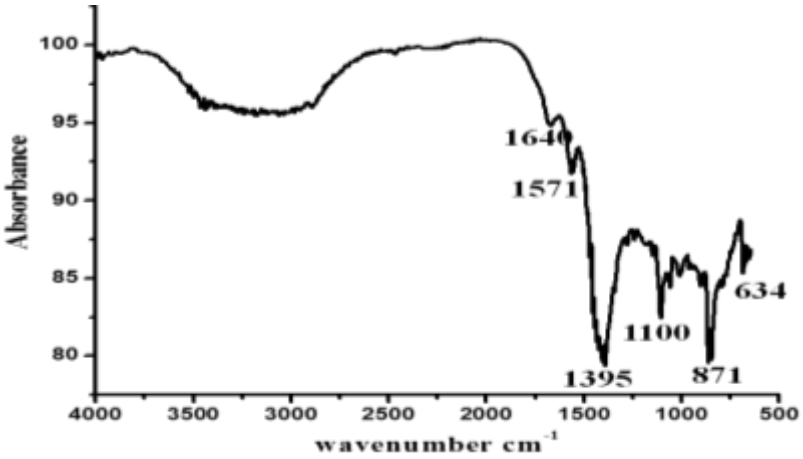

Fig. 2Fourier transform infrared spectra of PEG-CuONPs

X-ray diffraction measurements

The synthesized PEG-CuONPscharacterized by XRD data was illustrated in **Fig. 3**.The XRD pattern reveals that the intense and wide diffraction peaks at 2θ = 31.45º, 38.31º, 53.14° and 66.26° were attributed to planes of (110) (111) (202) and (022) of PEG-CuONPs crystalline structure. In addition, there are three less intense peaks at 2θ = 35.03º, 48.62º also observed, that can be ascribed to the (−111) and (-220) reflection ofthe CuO phase[24].**Fig.3** shows typical XRD patterns of the formedCuONPs which is identical to the JCPDS: 80-1916.The Sharp peaks were obtained atangles corresponding to the above planes indicates the monoclinic structure of PEG-CuONPs[25].No impurity peaks other than CuO wereobserved in the XRD pattern indicating the

high purity. The broadening of the diffraction peaks indicatesthat the crystal size is small. The average crystallitesize of the PEG-CuONPs calculated by Debye–Scherrer'sformula[26]as

$D = K\lambda / \beta\cos\theta$(1)

Where the constant K is taken to be 0.94, λ is the wavelength of X-ray (1.5406 Å), and β the full width at half maximum of the diffraction peak corresponding to 2θ. Using equation (1), theaverage size of synthesizedCuO nanoparticles was found to be 48 nm. It gives a single-phase with a monoclinic structure.

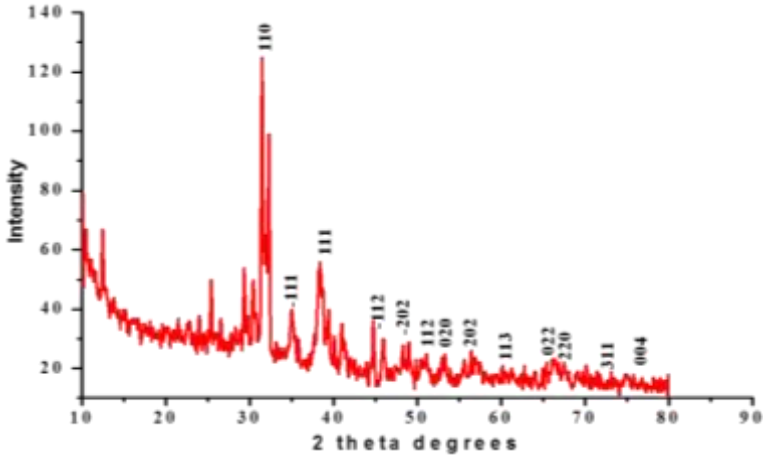

Fig. 3 X-ray diffraction spectra of PEG-CuONPs

Scanning electron microscope&Elemental dispersive spectroscopy

The **Fig. 4**shows the SEM image of synthesized PEG-CuONPsat various magnifications.SEM images show thesize of synthesized CuO nanoparticles is very small. The sizes of particles observed in SEM image are in the range of 30-70nm which is in good agreement with XRD spectral data. It shows that the particles are well crystallized and monoclinic in structure[27]. The EDS spectra of **Fig.5**shows two peaks corresponding to Cu and Oxygen molecules confirms the formation of PEG-CuONPs. Table inside the EDS spectra gives information about Cu atomic % was 50.51%, weight % was 18.97% and Oxygen atomic % was 28.23%, weight % was42.11%. The other elements were also observed may be from PEG.

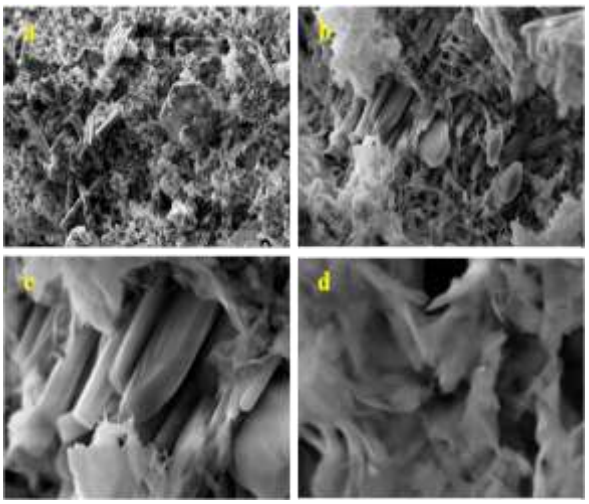

Fig. 4 Scanning electron microscopy images of the PEG-CuONPs

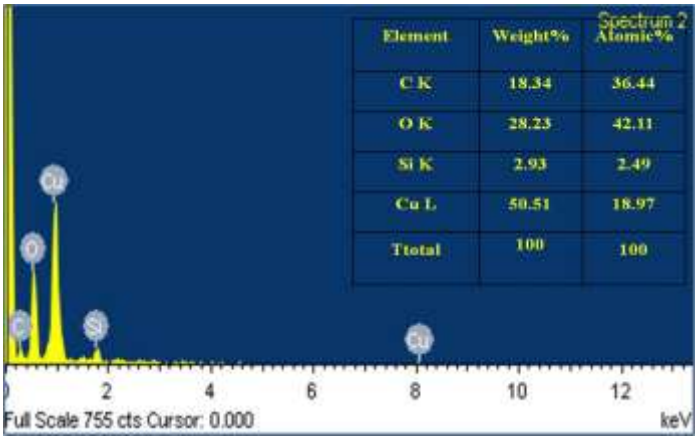

Fig. 5 Energy dispersive spectraof Cu and Oxygen elements of synthesized PEG-CuONPs and the table of EDS spectra.

Anti-bacterial activity

Cu can be used as aneffictive antimicrobial agent from the ancient times for various medical applications. The **Fig. 6**ofPEG-CuONPsexhibited antibacterial activity against all the three bacterial strains used in the study. Six various concentrations of PEG-CuONPs was added to the wells 1, 2, 3, 4, 5, 6 as 10µL, 20µL, 30µL, 40µL, 50µL, 60µL respectively and ampicillin used as control sample at centre of the plate which indicated as A. Clear zones of inhibition were seen although smaller zone of inhibition was found when compared to the standard antibiotic ampicillin. The results of zone of inhibition in mm for three different bacteria with respective samplesare shown in bargrapghof**Fig 7**.The Zone of inhibition for synthesized PEG-CuONPsat highest concentration for bacteria *Bacillus subtillis, Klebsiella pneumonia, Pseudomonas putida*, detected as 13 mm, 13 mm and 11 mm respectively.The Clear zone of inhibition was detected in the case of all three tested bacteria for 60µLwas more than that of control drug. Linear increasing of activity was observed with increasing concentrations,for *Pseudomonas putida*the activity observed only for50µL, 60µL concentrations.The proposed mechanism of antibacterial activity was the copper ions from PEG-CuONPs released and that they may attach to the negatively charged bacterial cellwall and damage it which leads to protein denaturation and cause cell death.On theother hand, the presence of capping agent PEG further enhances the cell death[28].

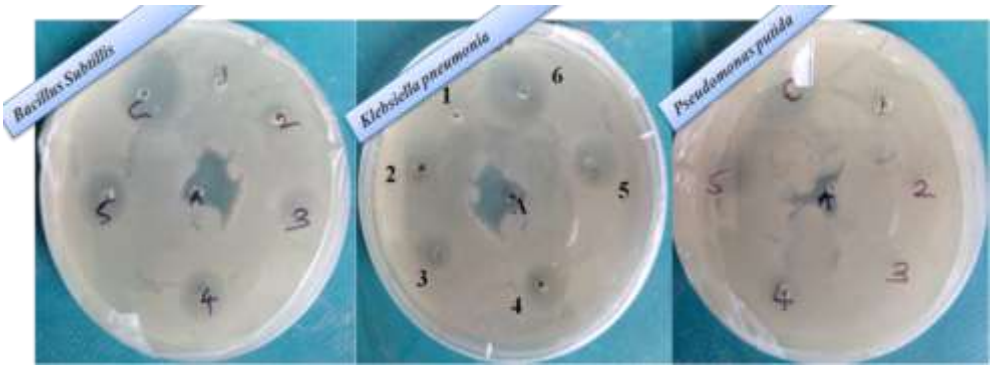

Fig. 6 Theantibacterial activity of PEG-CuONPsagainst three different tested bacteria such as *Bacillus subtillis, Pseudomonas putida, Klebsiella pneumonia.*

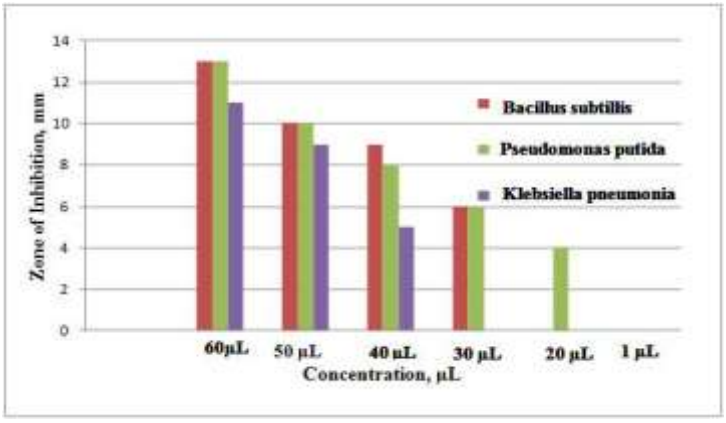

Fig. 7 The barchartof Zone of inhibition for three bacteria in mm

Conclusions

In view of the importance of transition metal oxide nanoparticles, we have successfully synthesized CuOnanoparticles by an aqueous precipitation method using Polyethylene glycol (PEG)as a capping agent and structurally characterized.UV-Vis spectroscopy confirms the formation of CuONPsand FTIR spectra supported that PEG was capped around the CuONPs. From The XRD spectral data monoclinic crystallite structure was confirmedwith the average crystallite size, 48 nm and good agreement with the SEM analysis. The elements present in the synthesized PEG-CuONPs were confirmed by EDS spectra. Further successfully investigated the antibacterial activity of PEG-CuONPs against *Bacillus subtillis, Klebsiella pneumonia, Pseudomonas putida*bacteria was proven to be effective activity. The present study concluded it constitute the basis for further investigations of the potential use CuONP as a chemotherapeutic.

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References

1. Fendler H.and Meldrum F.C., Adv.mater., 1995, 7, 607.
2. Lopez N,Japssens T.V.W, clause B.S, Xu Y, Mavrikakis M, Bligaard T.and Norskor J.K.,J. Catal., 2004, 223, 232.
3. Mitsuyu T, Yamakazi O,OhjiK. and WasaK, Ferroelectrics.,1982, 42, 233.
4. Regan O. and GratzelM, Nature., 1991, 353, 737.
5. Naazeeruddin K,Kay A. and Gratzel M, J. Am. Chem. Soc., 1993,115, 6832.
6. Bjoerksten U, Moser J. and Gratzel M, Chem. Mater., 1994, 6, 858.
7. Dow W.P. and Huang T.J, J. Catal. 1996, 160, 171.
8. Larsson P.O, Andersson A, Wallengerg R.L. and B. Svensson, J. Catal., 1996, 163, 279.
9. Lanje A.S, Ningthoujam R.S, Shrama S.J, VatsaR.K. and Pode R.B, Int. J. Nanotechnol., 2010, 7, 979.
10. Wolhfarth E.P.Ferromagnetic Materials, Vol. II,North-Holland, Amsterdam, New York, Oxford, Tokyo, 1980.
11. Jiang Y, Decker S, MohsC. and Klabunde K.J, J. Catal., 1998, 180, 24.
12. Lee S, Choi U.S, Li S and Eastman J.A, J. Heat Transfer., 1999, 121, 280.
13. RakhshniA.E. Solid State Electron.,1986, 29, 7.
14. Kumar R.V, Diamant Y. and Gedanken A, Chem. Mater,2000, 12, 2301.
15. Eliseev A.A, Lukashin A.V, Vertegel A.A, Heifets L.I, Zhirov A.I. and Tretyakov Y.D, Mater. Res. Innov.,2000, 3, 308.
16. Xu J.F, Ji W, Shen Z.X, Tang S.H, Ye X.R, JiaD.Z. and Xin X.Q, J. Solid State Chem., 2000, 147, 516.
17. Borgohain K, Singh J.B, Rama RaoM.V, ShripathiT.and MahamuniS, Phys. Rev., 2000, 61, 11093.
18. YuJ.Q, XuZ, JiaD.Z.and Chin. J. Func. Mater. Instrum., 1999, 5, 267.

19. Nakao M, Ikeyama T. Mizota P, Jin M, Tazawa Y Miyagawa S, Miyagawa S, and Wang L, Rep. Res. Cent., Ion Beam Technol., Hosei Univ. Suppl., 2000, 18, 153.
20. Yang C, Xiao F, Wang J,and Su X, J. Colloid Interface Sci.,2014, 435, 34.
21. Devi A.B, Moirangthem D.S, Talukdar N.C, Devi M.D, Singh N.R, and Luwang M.N, Chin. Chem. Lett.,2014, 25, 1615.
22. Vidyasagar C, Naik Y.A, Venkatesha T, and Viswanatha R, Nano-Micro Lett.,2012, 4, 73.
23. Perez C, Paul M. and Bazerque P., Act. Bio. Med.Exp., 1990, 15, 113.
24. Moralesa J, Sáncheza L, Martínb F, Jose R, Barradob R. and Sánchezb M, J. Electrochim. Acta.,2004, 49, 4589.
25. YaoW. T, Yu S. H, and Zhou Y, The Journal of Physical Chemistry. B.,2005, 109, 14011.
26. PattersonA. L.The scherrer formula for X-ray particle size determination,Physical Review, 1939, 56, 978.
27. Wang H, Zhang J, Zhu J. J.and ChenH.Y, J. Cryst. Growth.,2002, 244, 88.
28. Sondi I. and Salopek-Sondi B, J. Colloid Interface Sci.,2004, 275, 177.

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