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Synthesis of CuO Nanoparticles- A Chemical Reduction protocolE. JANARDHAN¹, M. MAHESHWAR REDDY¹, P. VENKAT REDDY¹ and M. JAIPAL REDDY*²

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Abstract

The present study has investigated the synthesis of copper nanoparticles by chemical reduction method without any inert gas. The nanoparticles obtained were characterized by UV-Vis, FTIR, XRD, and TEM spectroscopic techniques.

Key words: Copper oxide, XRD, SEM, Nanostructure, Precipitation.

Introduction

Reports on the different methods for synthesis of nanoparticles have shown the growing significance emotionally concerned to the synthesis of metal nanoparticles in the recent literature. Metal nanoparticles have plentiful industrial and scientific applications and are used for qualifying materials and instrument properties, improving human health etc. In this context, copper nanoparticles are commonly applied and many researchers have stressed the emphasis of copper nanoparticles properties, applications and synthesis. Previous studies on the thermal conductivity and viscosity of copper nanoparticles in fluids have shown an increase in the aforementioned properties versus the normal fluids. The measurements of some Cu suspensions indicate that the nanoparticle diameter, volume fraction, and bulk temperature have a considerable impact on the effective thermal conductivity of these nanofluids^{1,2}. It has been reported that copper nanoparticles show good catalytic activity both as homogeneous and heterogeneous catalytic hydroxylation of phenol by hydrogen peroxide to dihydroxybenzenes³⁻⁵. Copper nanoparticles show also antimicrobial activity, the starch-stabilized copper nanoparticles exhibited interesting antibacterial activity with both gram positive and gram negative bacteria at micro molar concentrations⁶. The copper nanoparticles have lethal effects against *Escherichia coli*, *B. subtilis*

and *S. aureus* microorganism^{7,8}. Furthermore, Copper nanoparticles are promising for application in cancer cell battling⁹.

A new green and efficient method has been developed for the synthesis of α -aminophosphonates using CuO¹⁰. Pure CuO nanocomposites were synthesized by using a modified perfume spray pyrolysis method (MSP)¹¹.

Hence, we herein report the synthesis of copper nanoparticles by chemical reduction method without any inert gas as a simple method. The materials are characterized by UV-Vis, FTIR, XRD, and TEM.

Materials and Reagents :

Copper (II) acetate (Cu(OAc)₂), copper (I) chloride (CuCl), copper (II) acetylacetonate (Cu(acac)₂), copper (I) per chlorate (CuClO₄), triethylsilane (Et₃SiH), octadecylsilate (ODS), tricylamine (TOA) and Octadecylamine (ODA) were purchased from Sigma-Aldrich and solvents toluene and ethanol were obtained from Rajadhani scientifics. All chemicals were used as received without further treatment. Freshly distilled toluene over Na/Benzophenone was used in the reactions.

Synthesis of copper nanoparticles:

Copper nanoparticles were prepared by reducing the copper (II) ions from copper (II) acetate dissolved in dry toluene in the presence of octadecylsilane. High molar concentrations; 10 equivalence of ODS is required to reduce the Cu⁺² ions due to its low standard oxidation potential. When 10 equivalents of ODS is added to the Cu (OAc)₂ solution in dry toluene at room temperature, the solution turned black color after 12 hrs indicates the formation of Cu nanoparticles which was further conformed by UV-Visible spectroscopy.

UV-Visible Spectroscopy:

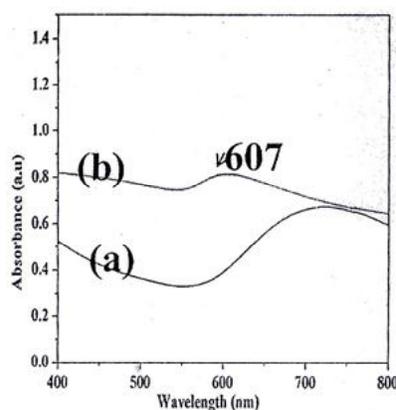


Fig. 1: UV-Visible Spectroscopy

A broad peak at 700 - 800 nm corresponds to Cu (OAc)₂ was disappeared by the reduction of Cu⁺² ions with ODS and a new peak at 607nm was formed due to the formation of Cu NPs. Here ODS acts both reducing as well as stabilizing agent.

Transmission Electron Microscopy (TEM):

TEM images shows that ODS forms a cross linked polymerized n-octadecylsilane micelles network and the different size of Cu NPs were embedded in that network.

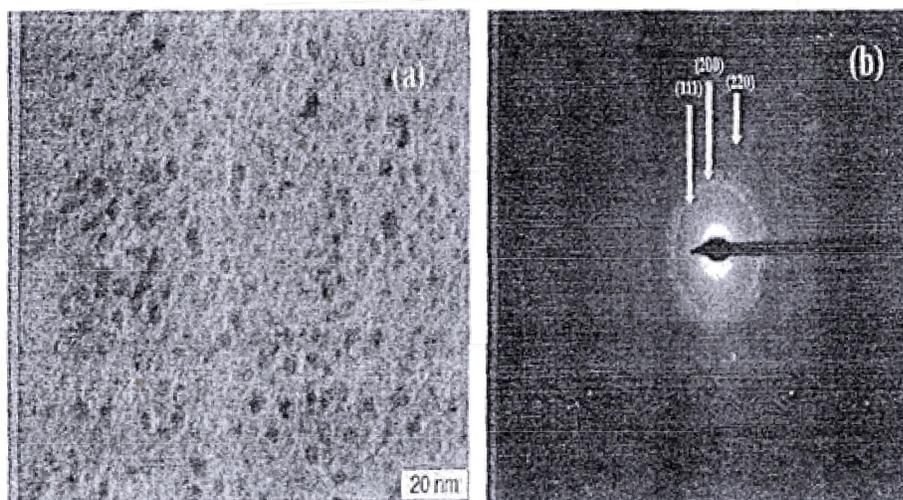


Fig. 2: Transmission Electron Microscopy

These particles were found to be stable in solution for up to 24 hrs but a significant amount precipitate was observed after 48 hrs of the storage of the reaction mixture. This observation indicated that these particles were highly active but susceptible to coagulation if stored for longer periods. The precipitate was analyzed by FTIR and XRD.

Fourier Transform Infrared Spectroscopy (FTIR):

The FTIR spectrum (Fig) of ODS shows prominent peaks at 2150 cm^{-1} , 2923 cm^{-1} and 2853 cm^{-1} corresponding to Si-H stretching, CH_2 asymmetric and CH_2 symmetric stretching vibrations of the groups present in ODS molecules. Another peak centered at 926 cm^{-1} can be assigned to the Si-O stretch of the silanol group, arising due to the partial hydrolysis of Si-H group on exposure to the atmosphere.

The peaks at 2150 cm^{-1} and 926 cm^{-1} from ODS disappear after the reaction and a new peak can be seen at 1024 cm^{-1} corresponding to the stretching mode of the Si-O-Si group. This silicates the oxidation of Si-H group to Si-O-Si.

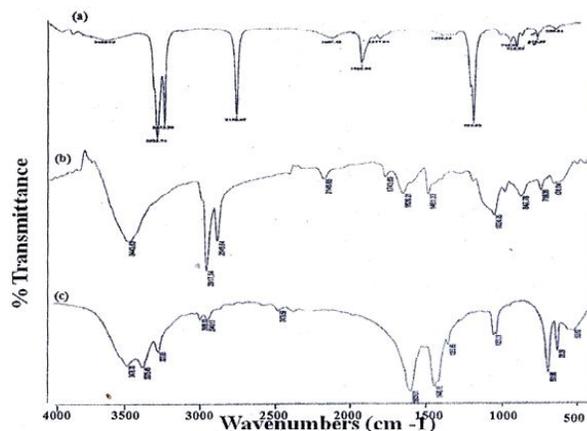


Fig. 3: Fourier Transform Infrared Spectroscopy

X-ray Diffraction (XRD):

XRD spectrum (Fig) shows a sharp, predominated peak at $2\theta = 21.33^\circ$ which corresponds to polymerized n-octadecylsilane micelles. Three main characteristic diffraction peaks for Cu were observed at around $2\theta = 43^\circ$, 50° , 74° correspond to (111), (200), (220) crystallographic planes of face centered cubic (FCC) Cu crystals (JCPDS no. 04-0784).

The lattice parameter “a” has been calculated by using these profiles and the average value of lattice parameter is found to be in agreement with reported value 3.615\AA . The crystallite size of Cu NPs is calculated using Eq.(1) and found to be around 10 nm which is in good agreement with the particle size measured from TEM images.

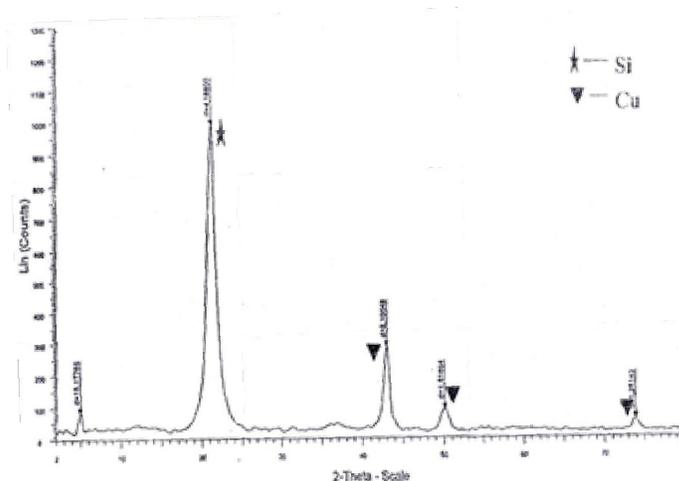


Fig. 4: XRD

When the reaction was conducted at 110°C with 1:10 molar ratios of $\text{Cu}(\text{OAc})_2$:ODS, the reaction mixture turned black within 15 minutes and the progress of formation of Cu NPs was monitored by UV-Visible spectroscopy. (Fig). TEM analysis shows that the Cu NPs were formed in different shapes with broad size distribution (Fig). The electron diffraction pattern of selected area (Fig) shows three debye-scherrer concentric rings assigned to (111), (200), and (220) consist with the face centered cubic (FCC) phase of the Cu NPs are large and the average diameter of the particles is in the range 2 - 12nm (Fig).

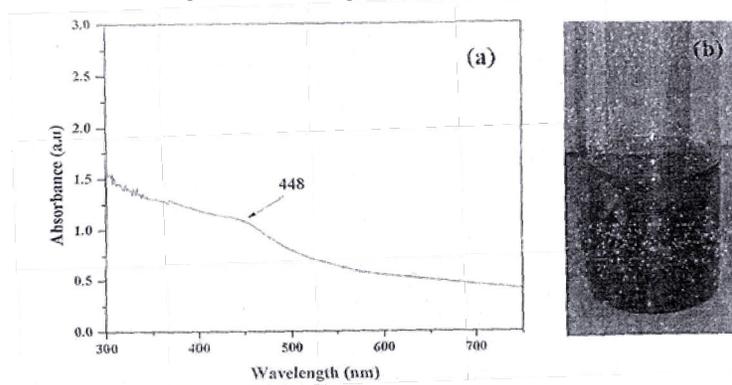


Fig 5: UV-visible spectrum

Though we have succeeded in the synthesis of Cu particles using ODS to reduce Cu ions, the particles need to be stabilized against oxidation. Amines are generally considered suitable for metal particles and also help to maintain a reducing atmosphere during reaction.

In this case, a long alkyl chain amine Trioctylamine (TOA) was chosen as stabilizing agent. When 4 molar equivalents of ODA was used as a capping agent only 4 molar equivalents of ODS was required to reduce Cu acetate in toluene at 110°C.

The reaction mixture turned yellow color and finally brown color and the resulting nanoparticles are stable in colloidal form. UV-Visible spectrum of the solution shows a peak at 448 nm.

It is clear that oleylamine has a considerable influence in limiting the growth as well as preventing the oxidation. These monodispers CuNPs were used for SERS applications.

Conclusion

We have successfully synthesized air stable colloidal Cu NPs by a simple chemical reduction method without any inert gas. The resulting nanoparticles were characterized by various spectroscopic techniques such as UV-Vis, FTIR, XRD, and TEM.

The formation of nano sized Cu was confirmed by its characteristic surface Plasmon absorption peaks in UV-Visible spectra. The transmission electron microscopic (TEM) images show that the resulting Cu nanoparticles are distributed uniformly with a narrow distribution. The X-ray diffraction (XRD) demonstrated that the obtained Cu NPs are single crystalline nanoparticles. Fourier Transform Infra-red (FTIR) spectroscopic data suggested that the silane Si-H is responsible for the reduction of Cu ions. The resulting colloidal Cu NPs were proved to serve as effective SERS-active substrates with SERS enhancement factors in the order of 10^3 .

Scope: These CuO nanoparticles can be used in synthesis of anti cancer agents, antioxidants as they are proven in lethal effects against microorganisms.

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