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MICROWAVE SYNTHESIS OF COPPER OXIDE NANOPARTICLES: OPTICAL AND STRUCTURAL CHARACTERIZATIONS

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Abstract : Copper oxide (CuO) nanoparticles have been synthesized by microwave assisted solvothermal method using copper acetate and urea as precursors. This method gives a large scale production of CuO nanoparticles easily.The particle sizes have been obtained as, 10-14 nm. The synthesized pure copper oxide nanoparticles have been characterized by using XRD, SEM–EDAX ,UV - Visible and VSM analysis techniques. X-ray diffraction pattern (XRD) reveals single phase monoclinic structure. Scanning electron microscopy (SEM) showed the spherical morphology of as prepared CuO nanoparticles. The UV-Visible absorption spectrum of CuO nanoparticles shows a strong blue shift compared to that of bulk, The antiferromagnetic (AFM) nature of CuO nanoparticles are studied using vibrating sample magnetometer (VSM).

Keywords : Nanoparticles, solvothermal, Copper oxide, antiferromagnet.

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1. Introduction

The oxides of transition metals are an important class of semiconductors, which have applications in magnetic storage media, solar energy transformation, electronics and catalysis [1-9] 1,4,7,9,10,12,13,16,18. Among the oxides of transition metals, 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 % [10] [11].CuO is a semiconducting compound with a narrow band gap and used for photoconductive and photothermal applications [11] [15]. However, the reports on the preparation and characterization of nanocrystalline CuO 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 nanocrystalline CuO have been reported recently such as the sonochemical method [12][8], sol–gel technique [13][5], one-step solid state reaction method at room temperature [14][19], electrochemical method [15][2], thermal decomposition of precursors [16][20] and co-implantation of metal and oxygen ions [17][14] and so on. In the present work, we have synthesized and investigate size, morphology, optical and magnetic properties of the phase pure CuO nanoparticles by a simple microwave assisted solvothermal method .

2. Methods and Materials

The present work is focused on the synthesis and characterization of nanometer-sized pure CuO particles by a simple microwave assisted solvothermal method. Analytical reagent (AR) grade copper acetate, urea, ethylene glycol (as solvent) are used as precursors. Copper acetate and urea are taken as solute in the molecular ratio 1:3 and dissolved in 100 ml ethylene glycol as individually. The prepared solution is kept in a domestic microwave oven (operated with frequency 2.45 GHz and power 800W). Microwave irradiation is carried out till the solvent is evaporated completely. The obtained colloidal precipitate is black in colour. In the end, acetone washing is used to remove the organic impurities. The prepared sample is dried in atmospheric air and annealed it for 30 minutes at 100^{0}C to improve the ordering. The reaction time, yield percentage and color of the samples are noted. The synthesized NPs have been characterized by using X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), EDAX, UV-visible absorption, and Vibrating Sample Magnetometer (VSM).

2.1 Characterization

The powder X-ray diffraction (XRD) was performed using automated X-ray diffractometer (X-PERT PRO Philips System) operating CuK_{α} at wavelength 1.54056 Å. The average crystallite size (D) has been calculated using Scherer's relation $D = K\lambda/\beta\cos\theta$, where the constant K is taken to be 0.94, λ is the wavelength of X-ray used , where β the is full width of half maximum (FWHM). The morphology of the powder samples was characterized by scanning electron microscope (SEM) JEOL/EO JSM-6390. The UV spectrum was obtained using LAMBDA-35 UV visible spectro photo meter. The antiferromagnetic (AFM) nature of CuO nanoparticles are studied using vibrating sample magnetometer (VSM).

Results and Discussion

3.1 XRD Analysis

XRD pattern of as prepared CuO nanoparticles is shown in Figure 1. It gives a single-phase with a monoclinic structure. The lattice parameters are calculated using the least square refinement from the UNITCELL-97 program [18][6]. The obtained parameters are a= 4.682 Å, b= 3.424 Å, C=5.127 Å with volume cell of 81.52 $Å^3$. These values are consistent with the reported literatures 13 17 [19,20] and with the respective "JCPDS" (Joint Committee on Powder Diffraction Standards) card No.89-5895.No peaks of impurities are found in XRD pattern. The peaks are broad due to the nano-size Effect. The average crystallite size of CuO nanoparticles is found to be 10-14 nm using Scherrer formula.

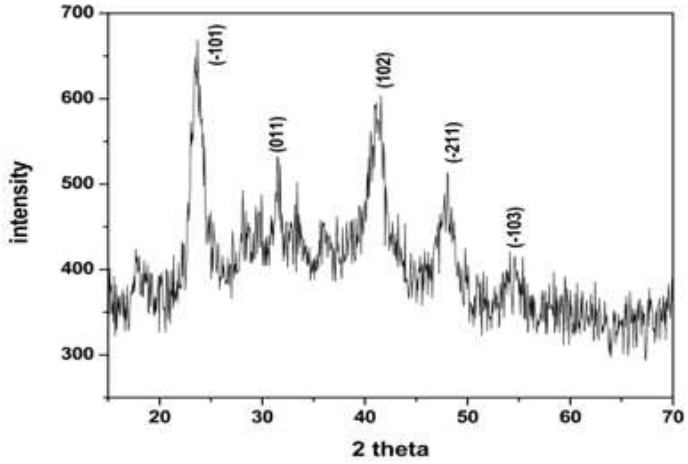

Figure.1.PXRD spectrum for pure CuO nanoparticles

3.2 Structural Studies

Figure 2 shows the SEM image of as prepared CuO nanoparticles. The size and morphology of CuO nanoparticles have been examined by SEM. It shows that the CuO nanoparticles are in spherical shape. SEM micrographies clearly show the surface features, by which it highlight that CuO nanoparticle was successfully prepared and it can be seen that the particles congregate together and the size of which is about 100 nm. The doping levels and the bonding characteristics are determined by EDAX spectrum. The EDAX spectra of CuO nanoparticles are shown in Figure 3. This shows the elements of crystal matrix CuO.

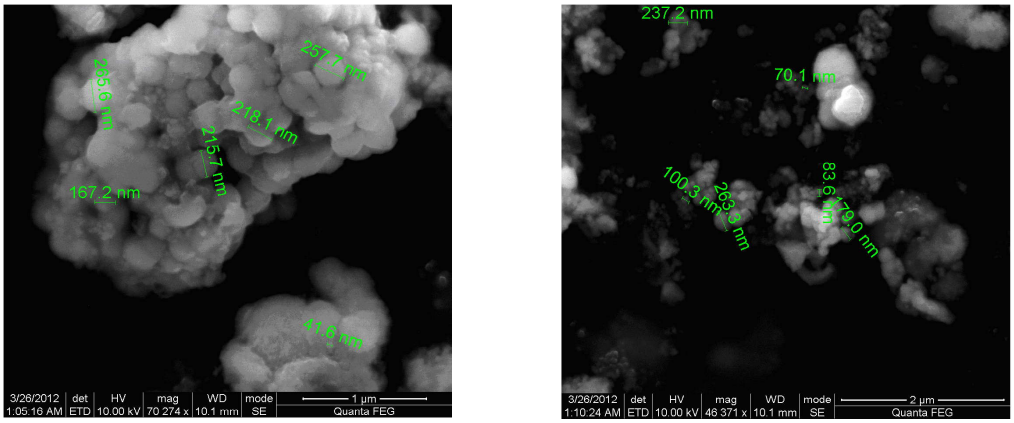

Figure 2. SEM images for pure CuO nanoparticles

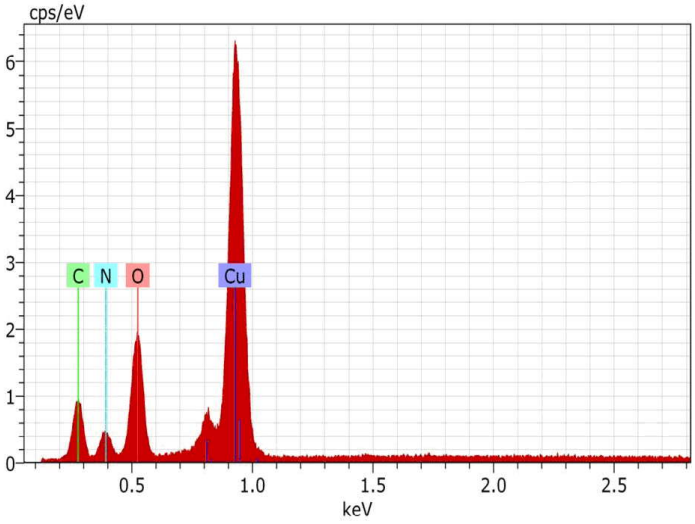

Figure 3. EDAX image for pure CuO nanoparticles

3.3 Optical Studies

UV-Visible absorption and reflection spectrum for pure CuO nanoparticles are shown in Figure 4. The spectrum shows the band edge-absorption peak which is found to be at 270 nm. In UV-Vis, high energy electromagnetic radiation in the wavelength range of 100-700nm is utilized to promote electrons to higher energy orbital's. From the UV spectra, it is clear that the absorbance decreases with increase in wavelength. This decrease in the absorption indicates the presence of optical band gap in the material.

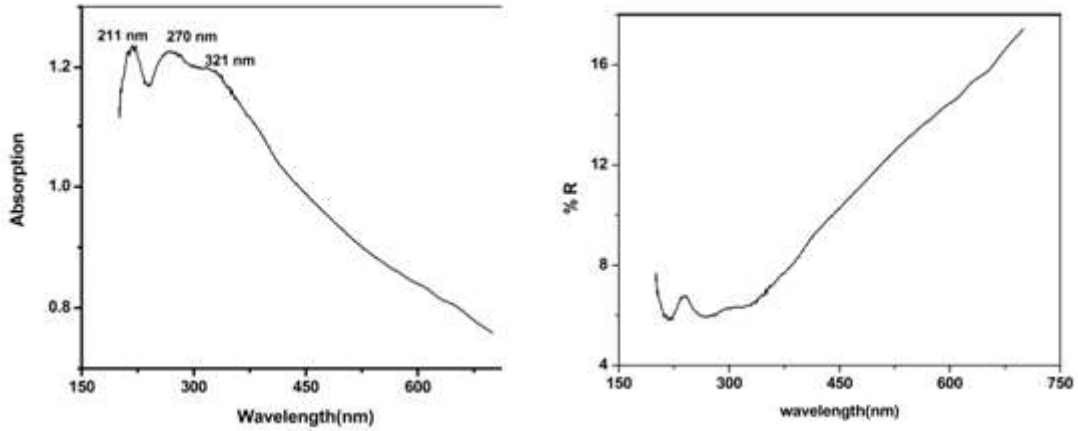

Figure 4. UV-Visible absorption and reflection spectrum pure CuO nanoparticles

3.4 Magnetic Studies

Hysteresis cycles measured at 300 K are shown in Figure 5. The magnetizations of the three samples increase with increasing magnetic field strength. It is also observed that at smaller magnetic fields the magnetizations increase with magnetic field nonlinearly whereas at relatively higher magnetic fields the magnetizations increase with magnetic field almost linearly. These are characteristics of antiferromagnetic materials.

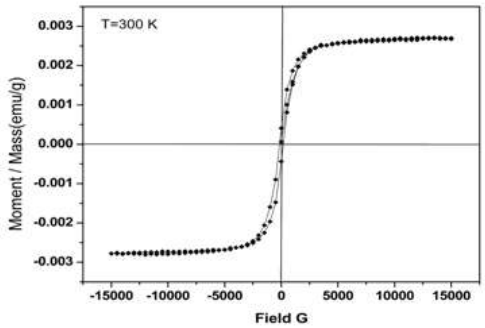

Figure 5. VSM spectrum for pure CuO nanoparticles

4. Conclusion

CuO nanoparticles with monoclinic structure are synthesized successfully by microwave assisted solvothermal method. SEM micrographies clearly show the surface features, by which it shows that the CuO nanoparticles was successfully prepared and it can be seen that the particles congregate together and the size of which is about 100 nm. EDAX spectra show the elements of crystal matrix CuO. From the UV spectra, it is clear that the absorbance decreases with increase in wavelength. This decrease in the absorption indicates the presence of optical band gap in the material. The band-edge emission peak is found at 270 nm.VSM spectra shows the antiferromagnetic nature of CuO nanoparticles.

References

[1] U. Bjoerksten, J. Moser, M. Gratzel, Chem. Mater. 1994, 6, 858.

[2] K. Borgohain, J.B. Singh, M.V. Rama Rao, T. Shripathi, S. Mahamuni, Phys. Rev.,2000, 61, 11093.

[3] Daqiang Gao, Jing Zhang, Jingyi Zhu , Nanoscale Res Lett. 5 (2010) 769–772.

[4] W.P. Dow, T.J. Huang, J. Catal., 1996,160, 171.

[5] A. A. Eliseev, A.V. Lukashin, A. A. Vertegel, L.I. Heifets, A. I. Zhirov, Y. D. Tretyakov, Mater. Res. Innov., 2000, 3, 308.

[6] T.J.B. Holland, S.A.T. Redfern, Miner. Mag. 61 (1997) 65.

[7] Y. Jiang, S. Decker, C. Mohs, K.J. Klabunde, J. Catal. 1998, 180, 24.

[8] R.V. Kumar, Y. Diamant, A. Gedanken, Chem. Mater., 2000, 12, 2301.

[9] A.S. Lanje, R.S. Ningthoujam, S.J. Shrama, R.K. Vatsa, R.B. Pode, Int. J. Nanotechnol., 2010, 7, 979.

[10] P.O. Larsson, A. Andersson, R.L. Wallengerg, B. Svensson, J. Catal., 1996, 163, 279.

[11] S. Lee, U.S. Choi, S. Li, J.A. Eastman, J. Heat Transfer, 1999, 121, 280.

[12] T. Mitsuyu, O. Yamakazi, K. Ohji, K. Wasa, Ferroelectrics, 1982, 42, 233.

[13] K. Naazeeruddin, A. Kay, M. Gratzel, J. Am. Chem. Soc., 1993, 115, 6832.

[14] S. Nakao, M. Ikeyama, T. Mizota, P. Jin, M. Tazawa, Y. Miyagawa, S. Miyagawa, S. Wang, L. Wang, Rep. Res. Cent. Ion Beam Technol., Hosei Univ. Suppl., 2000, 18, 153.

[15] A.E. Rakhshni, Solid State Electron., 1986, 29, 7.

[16] O. Regan, M. Gratzel, Nature, 1991, 353,737.

[17] D.P. Volanti, D. Keyson, L.S. Cavalcante et al, Journal of Alloys and Compounds 459 (2008) 537-542.

[18] E.P. Wolhfarth; Ferromagnetic Materials, Vol. II, North-Holland, Amsterdam, New York, Oxford, Tokyo, 1980.

[19] J.F. Xu, W. Ji, Z.X. Shen, S.H. Tang, X.R. Ye, D.Z. Jia, X.Q. Xin, J. Solid State Chem., 2000, 147, 516.

[20] J.Q. Yu, Z. Xu, D.Z. Jia, Chin. J., Func. Mater. Instrum., 1999, 5, 267.