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Canted antiferromagnetism in copper oxide nanoparticles synthesized by the reverse-micellar route

T. Ahmad [a], R. Chopra [a], K.V. Ramanujachary [b], S.E. Lofland [b], A.K. Ganguli [a,*]

[a] *Department of Chemistry, Indian Institute of Technology, Delhi, New Delhi 110016, India*
[b] *Department of Chemistry and Physics, Center for Materials Research and Education, Rowan University, 201 Mullica Hill Road, Glassboro, NJ 08028, USA*

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Abstract

Copper oxide nanoparticles were synthesized by water-in-oil microemulsions with two different apolar solvents (isooctane and *n*-octane). Our studies on the samples show that the grain size is highly dependent on the nature of non-polar solvent, 25–30 and 80–90 nm sized nanoparticles with isooctane and *n*-octane, respectively. The Neel temperature of CuO nanoparticles obtained from isooctane is about 80 K. For the larger particles obtained from *n*-octane, the transition temperature shifts to higher temperature (∼ 220 K) near that of the bulk.

Keywords: Cu oxide; Magnetic nanoparticles; Canted antiferromagnetism; Chemical synthesis

1. Introduction

Nanoparticles and nanostructured materials are among the most challenging areas of current scientific and technological research. Nanoparticles have technological applications in areas such as catalysts, materials processing, and high performance ceramics. Many physical properties of nanoparticles often differ drastically from those of single crystals with the same chemical composition.

Numerous processes are being developed aiming at the synthesis of nanoparticles of a variety of materials. Among them, solvothermal [1,2], polymeric-precursor [3] and surfactant-mediated routes [4–7] are quite popular. The surfactant-mediated route with reverse micelles is of special importance since it provides homogeneous and monodisperse nanoparticles without the need for specialized or expensive equipment. The reverse-micelles obtained at a particular ratio of the aqueous phase to the surfactant leads to uniform-size nanoreactors and have an aqueous core [8,9] in which it is possible to precipitate the inorganic material. In most cases a precipitating agent such as carbonate, oxalate or an organic ligand is allowed to react with the metal ion, and the resulting precipitate yields the oxide of choice upon decomposition.

Copper oxide nanoparticles are of special interest because of their efficiency as nanofluids in heat-transfer applications. For example, it has been reported that 4% addition of CuO improves the thermal conductivity of water by 20% [10]. Of equal importance from a fundamental viewpoint is the magnetism of the Cu(II) ions in various environments because of its relevance to high-temperature superconductivity in several CuO-based ceramics [11] and dilute Cu (II) systems have been investigated for low-dimensional magnetic correlations [12–14].

In this paper we report for the first time the synthesis of CuO nanoparticles by a reverse-micellar route. We have characterized the samples by X-ray diffraction, transmission electron microscopy and field-dependent magnetization studies.

* Corresponding author. Tel.: +91-11-26591511; Fax: +91-11-26854715.
E-mail address: ashok@chemistry.iitd.ernet.in (A.K. Ganguli).

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2. Experimental

The synthesis of the CuO nanoparticles was carried out in two steps. First, we synthesized the nanoparticles of copper oxalate from two microemulsions (**I** and **II**) as described below. In the second step, the copper oxalate particles were subjected to a careful thermal decomposition to yield nanoparticulate CuO.

Microemulsion **I** is composed of cetyltrimethylammonium bromide (CTAB) as the surfactant, *n*-butanol as the cosurfactant, isooctane (or *n*-octane) as the hydrocarbon phase and 0.1 M copper nitrate solution as the aqueous phase. Microemulsion **II** has the same constituents as above except for having 0.1 M ammonium oxalate instead of copper nitrate as the aqueous phase. The weight fractions of various constituents in these microemulsions are follows: 16.76% of CTAB, 13.90% of *n*-butanol, 59.29% of isooctane and 10.05% of the aqueous phase. These two microemulsions were mixed slowly and stirred overnight on a magnetic stirrer, and the resulting precipitate was separated from the apolar solvent and surfactant by centrifuging and washing it with 1 : 1 mixture of methanol and chloroform. X-ray diffraction data of the decomposed product was collected with a Bruker D8 advance diffractometer using Cu-Kα ($\lambda = 1.5418$ Å) radiation. The lattice parameters were calculated using a least-squares fitting procedure of the observed d values. Transmission electron microscopic (TEM) studies were carried out using a JEOL JEM 200CX electron microscope operated at 200 kV. TEM specimens were prepared by dispersing the powder in acetone by ultrasonic treatment, dropping onto a porous carbon film supported on a copper grid, and then dried in air.

On the basis of the thermogravimetric studies, we calcined the precursor (obtained by the reverse micellar route) at 450 °C for 6 h to effect the conversion to CuO nanoparticles. Two sets of CuO nanoparticles were obtained by the above procedure, one where isooctane was used as the nonpolar medium (oil) and another where *n*-octane was used as the oil to form the reverse micelles.

The magnetization was measured at temperatures ranging from 5 to 300 K, in applied fields of up to 5000 Oe with a Quantum Design Physical Properties Measurement System.

3. Results and discussion

The powder X-ray diffraction pattern of the CuO particles is shown in Fig. 1. All the reflections in the pattern could be indexed on the basis of a monoclinic cell reported for copper oxide (JCPDS # 48-1548). The refined unit cell para-

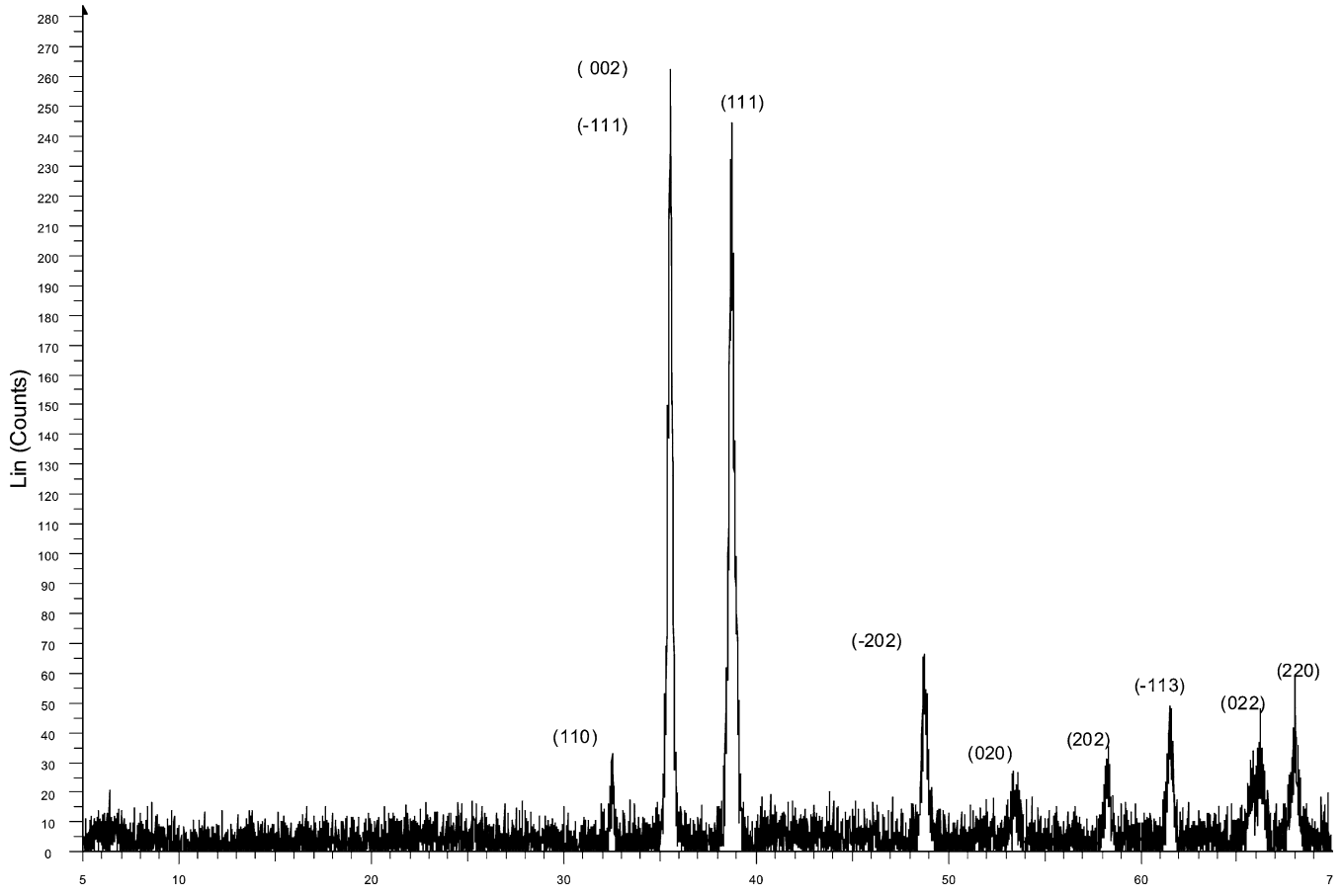

Fig. 1. Powder X-ray diffraction pattern of CuO nanoparticles prepared by reverse micellar route.