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Short Communication

Simple chemical method for synthesis of CuO/CuI nanocomposite

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ABSTRACT

This study reports the simple process to synthesis of CuO/CuI nanocomposite in a non-aqueous solution. In the conducted experiments CuI and Dimethyl Sulfoxide (DMSO) were used as the precursors. Moreover, DMSO had role of solvent and ligand simultaneously. In experiments the effects of oleic acid as the particles size modifier, temperature and concentration were investigated. Crystalline structure, size and shape of the CuO/CuI nanocomposite were characterized using X-ray diffraction (XRD), and scanning electron microscopy (SEM) techniques, respectively. The results indicated that particles size is less than 50 nanometers and are spherical and mono-dispersed in shape and size. In addition, the Crystallite sizes were estimated via the Scherrer equation.

Keywords: *XRD; CuO/CuI; SEM; X-ray; Nanocomposite.*

INTRODUCTION

Nano-scale materials have important roles both in fundamental and practical research areas due to theirstable chemical and physical properties as well as favorable applications in nanodevices [1]. Decrease of the particle size to nanodimensions results in diverse excellent properties in comparison to their mass properties[1]. CuO is a p-type semiconductor with a thin band gap (1.2 Ev)[2],used for photoconductive and photothermal applications. In addition, some groups informed that CuO can be presented in a variety of three diverse magnetic phases [3].Moreover, CuO has been used as heterogeneous catalysts in many important chemical procedures, such as oxidation of carbon monoxide, hydrocarbon and phenol in supercritical water, and degradation of nitric oxide, selectiveand catalytic reduction of nitric oxide with ammonia [2].

Numerous approaches have been used for the fabrication of nanosized CuO (including Cu_2O), such as solid-state reaction procedure[4],alcohol thermal decomposition of copper acetate [5], sonochemical method [6], direct oxidation of copper substrates in air at high temperature[7], hydrothermal decomposition route [8], sol–gel method [9], solid–liquid phase arc discharge process[10] electrochemical synthesis [11] and so on.

Cupric iodide (CuI), as a p-type semiconductor and with a wide and direct band gap (more than 3eV) in the zinc-blend in basic -state phase, is a promisingmaterial for creating short wavelength emitters and formakingtransparent optoelectronic devices. Recently, significant research attempts, both experimentally and theoretically, hasbeen devoted to the study of the band structure and transport virtues of CuI [12].

Since Tennakone *et al.* reported the utilization of CuI as a hole-collecting factor in a dye-sensitized solid state cell [13],room-temperature blue-emitting devices, field emission displays, and vacuum fluorescent displays. Muchconsideration havebeen concentrated on studying the properties of CuI, containing thin CuI filmsand Cuquantum dots.Quantum size effects in CuI nanocrystals embedded in glass have been investigated experimentally, and uncommon luminescence behavior has been observed, includingluminescence elongation followed by the increase in the light exposure [13-17]. Besides, CuI is also a traditional and important catalyst for synthesis of many organic compounds[18], solid state X-ray and g-ray detectors andsolid electrolytes in solid state batteries [19, 20]. So far, several physical and chemical methods such as pulse laser deposition [21], Hybrid electrochemical/chemical synthesis [22]and water-in-oil (w/o) microemulsions [23] have been used to synthesize CuI nanostructures. Also, recently Kozhummal *et al.* [24] reported a facile high-yield formation of CuI superstructures by antisolvent crystallization with and without organic additives.

In this paper, for the first time we provide a simple method to synthesize monodispersed CuI/CuO nanocomposite from a solution precursor using CuI and Dimethyl Sulfoxide (DMSO) without applying any complexapparatus and reagents. It is a simple andeffective method to synthesize CuI/CuO nanocomposite in large quantity a balanced temperature.We expect in near future there will founded new applications for this composite.

EXPERIMENTAL

Materials and Techniques

Copper iodide (I) (laboratory degree) and Oleicacide had been purchased from Merck, and DMSO was purchased from Sharlo. The field emission scanning electron microscopy (FE-SEM) images were obtained using Hitachi S4160 scanning electron microscope. The X-ray diffraction patterns of the products were recorded by employing an INEL X-ray diffractometer (model Equinox 3000).

Methods

CuO–CuI nanocomposite in DMSO (Figure 1) was synthesized by following method: After dissolving 3 g CuI in 30 mL DMSO, the solution was heated above 80 °C under constant stirring rate. The grey solution turned black and after a few minutes the particles were precipitated at bottom of the experiment dish. The resultant black products were washed thoroughly with distilled water and dried at ambient condition. To investigate of surfactant effects on the morphology and particles size, in second experiment 0.1 ml oleic acid was added into solution then the above experiment was repeated. In continue to investigate the influence of the concentration and reaction temperature on the morphology and size of the products; we conducted two more experiments to compare the results. To examine the effects of concentration on the products properties in the third experiment 60 ml DMSO was used for dissolving 3 gr CuI. Finally, in order to investigate temperature effect, the forth experiment was performed at 120°C while the solution concentration was equal to the concentration in the third experiment.

RESULTS AND DISCUSSION

Figure 1 represents the X-ray diffraction pattern related to the synthesized CuO/CuI sample at the environment atmosphere. Obviously, it is

shown in this pattern that the solid sample consisted of both CuO and CuI phase. In XRD pattern the samples index to tenorite, synCuO (JCPDS number 00-045-0937) and CuI (JCPDS number 00-083-115) although have different intensities of crystallinity. From this figure, the material has an ordered crystalline structure. There are five main diffraction peaks in the pattern which the two main diffraction peaks at 35.5° 2θ and 38.3° 2θ are belong to tenorite, synCuO with monoclinic structure and the others at 25.32°,41.97° and 49.7° are belong to CuI with cubic structure . It seems, from pattern fluctuations, that CuI has a more crystallinity level than CuO. Crystallite sizes were estimated via the Scherrer equation; i.e.,

$$d=\frac{0.9\lambda}{\beta \cos \theta}[25].$$

Where d is the crystallite thickness (nm) in the direction vertical to the diffraction plane, λ is the wavelength of radiated X-ray radiation (nm), β is the full-width half maximum of the peak (rad), and θ the diffraction angle (rad). Thecoefficient0.9 is dimensionless shape factor, and is the most generally used value. For the (111) peak (~25° 2θ) and for the (111) peak (~38.2° 2θ) the crystallite thicknesses are 45 and 5.8 nm for CuI and CuO, respectively.

Figure 2 shows the SEM images of the synthesized samples in variety of conditions in the experiment. The images indicate that all particles are almost spherical in shape and smaller than 50 nm in size. From the SEM image of the synthesized particles at 80 °C (Figure 2a), which in their synthesis process no surfactant was used, one can tell that boundaries of particles are clear. Figures 2b and 2c are the SEM image of two samples which in their synthesis process oleic acid was used. These images show that the particles are agglomerated in the texture of surfactant (oleic acid). From the SEM images there are no considerable differences between the morphology of the particles. Butone can tell that the particles size in Figure 2b are bigger than particles size in Figure 2c which indicates the effect of lower solution concentration on reducing the particles size. The SEM images in Figure 2d showsthatdue to high temperature synthesis,the agglomeration of particles in the surfactant texture havebeenreduced in comparison with Figure 2c.

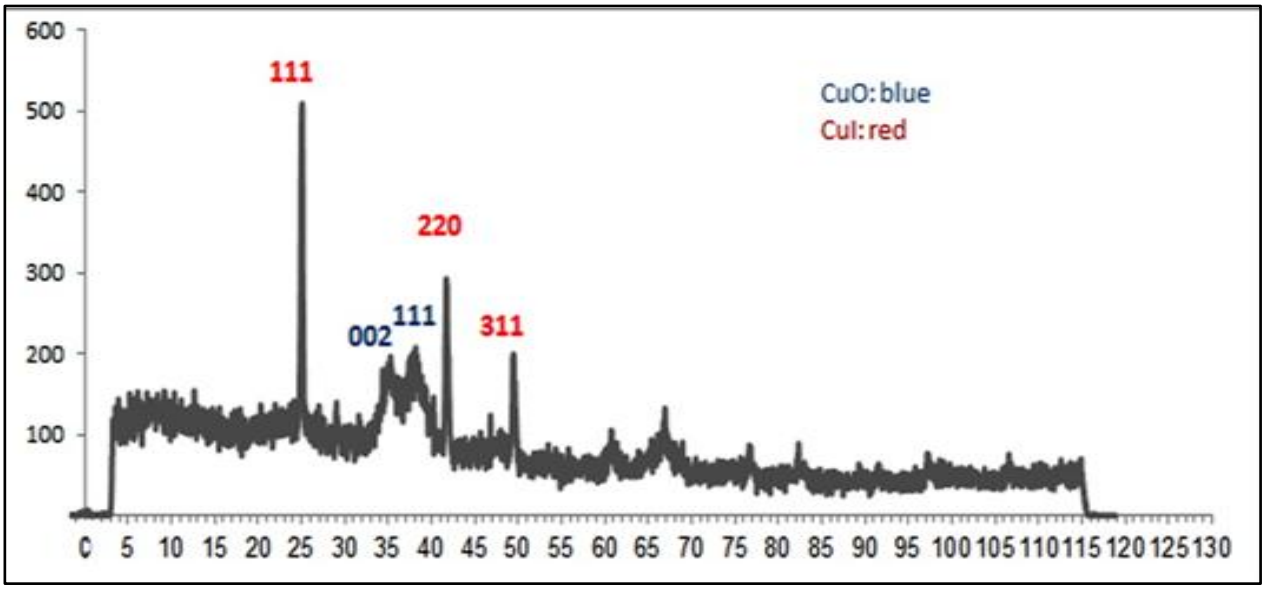

Fig. 1. XRD pattern of CuO/CuI nanocomposite.

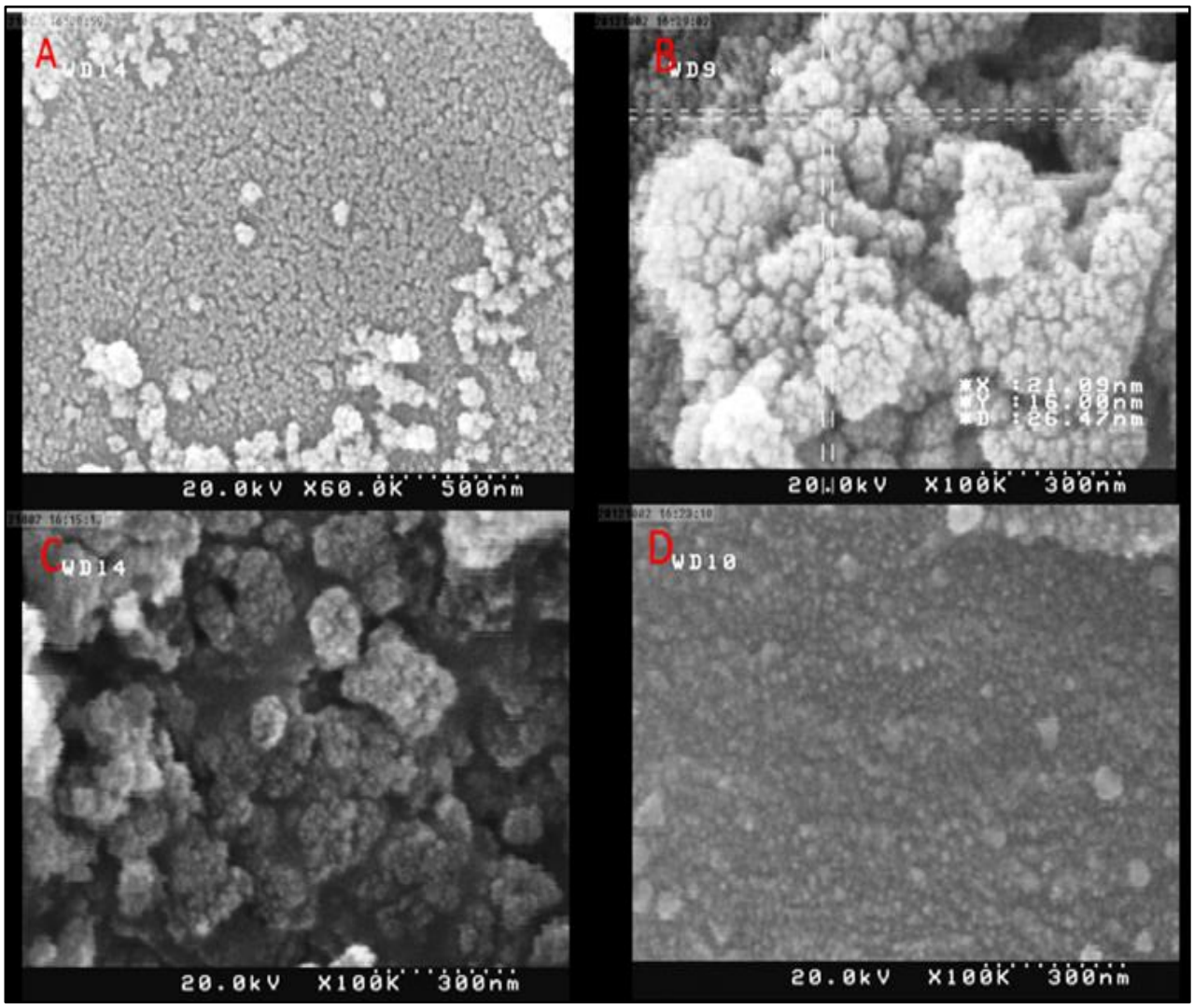

Fig. 2. SEM images of CuO/CuI nanocomposite at various reaction condition: A)at 80°C B) at 80°C in presence of oleic acid C) at 80°C low concentration D) at 120°C and low concentration.

In this work, after preparation of Cu^{+} ion at mean temperature, in order to progress the reaction temperature was increased. A DMSO molecule has three lone electron pairs, two of which are on the oxygen atoms and one is on the Sulfur atom. Cu^{+} (d^{10}) ion is a soft acid which prefers sulfur lone pair rather than oxygen lone pairs. This ion is stabled by the bonds between DMSO lone pairs and its unoccupied orbitals. In fact, DMSO properties led to formation of Cu^{+} ion needed to reaction. Because of the stability of this complex, reaction hadn't taken place at room temperature, but after heating the solution above 80^{0} C the products were manufactured.

CONCLUSIONS

A simple novel chemical approach to prepare CuI/CuO nanocomposite is reported. The results show that non- aqueous solvent is suitable to produce tiny Copper-based composites. Copper (I) ion is stable in DMSO.So this solvent is appropriatefor copper –based material treatments. This method may be used in industrial and commercial applications because of its rapid

reaction and cheap materials to apply. Simplicity and fast functions are advantages of this process.

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