



A facile, green, one pot synthesis of cuprous iodide nanoparticles using the mechanochemical method



Saeed Shahbazi, Shahrara Afshar*

Department of Chemistry, Iran University of Science and Technology, 16846-13114 Tehran, Iran

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ABSTRACT

Cuprous iodide, that recently has been used as an inorganic hole (vacant state) conductor material in dye sensitized solar cells, was successfully synthesized with a rapid, low cost, one pot and solvent free reaction using the mechanochemical method. The XRD data showed that all peaks matched well with those of the gamma phase of CuI. In addition, EDX analysis confirmed the formation of 100% pure CuI compound. Moreover, the SEM images showed the nanosized spherical morphology of 40–70 nm. The diffuse reflectance spectrum (DRS) of the product showed that CuI nanoparticles have a maximum absorption at about 400 nm and a band gap of 3 eV. The results of this contribution corroborate the successful synthesis of pure cuprous iodide (CuI) nanoparticles.

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1. Introduction

Cuprous iodide (CuI) has many features that derived much attention during the last decade. It has large band gap, an unusually large temperature dependency and large ionicity [1,2]. This compound has also been applied as a potent catalyst for organic compound syntheses [3], ultrafast scintillators [4], and super ionic conductors, solid-state solar cells, etc. [5].

One of the main components of dye solar cells is electrolyte. Iodine-triiodide liquid electrolyte is a composition frequently used in dye solar cells [6]. However despite having a good performance as liquid electrolytes, it suffers from drawbacks such as volatility, corrosion and sealing problems that decrease the stability of the cells [7]. Hence, replacing the liquid electrolyte with solid electrolytes is one of the major challenges in producing of dye solar cells. Solid electrolytes among others have attracted much attention due to their high stability, high hole transfer speed and physical contact at the interface of a metal oxide/dye/electrolyte [7]. CuI and CuSCN are used as solid electrolytes in dye sensitized solar cells since a good overlap exists between the bands of CuI and dye [8,9].

Cuprous iodide has been prepared using several physical methods such as electrodeposition [10], pulse laser deposition [11], vacuum evaporation [12]. And different chemical methods have been reported on preparing CuI, such as the liquid phase reaction [13], low temperature solvothermal synthesis [5], coprecipitation [14], and precipitation method using capping agent and pomegranate juice [15,16]. Although the above mentioned

methods may have some advantages and despite significant gains in the past, still finding a new route for green, facile and scale-up preparation of the cuprous iodide remains a challenge.

In recent years, numerous scientists and technologist have paid much attention to solid state reactions because of their simplicity of application in large scale, low-cost and high efficiency. Mechanochemical processes are used to prepare different kinds of nanostructures of metal oxides [17], hydrogen storage materials [18], composites [19] and so on. Since no toxic liquid solvent have been used in these reactions, they do not pollute the environment and therefore these reactions can be considered as green chemistry methodologies. The above advantages have made the researchers to be high concerned about the mechanochemical methods compared to the other synthetic routes.

In this work, for the first time, a mechanochemical method is used for the synthesis of the spherical γ -CuI nanoparticles at room temperature. This study introduces a facile, green and an easiest possible one-pot method for the synthesis of CuI compound. The proposed method has solvent-free characteristic which would result in a lower cost and being environmentally favored. In addition, the procedure of CuI synthesis is very easy in the presented work.

2. Material and method

CuSO₄ (99%) and KI (99.5%) were purchased from Merck Company and were used without further purification.

In a stainless steel (10 mL) vial a mixture of CuSO₄ and KI solids with a molar ratio of 1:2 were milled together using a Mixer Mill (Retsch MM-400) apparatus at 1800 rpm (30 Hz) for 40 min at room temperature. The brown powder as a product was expected

* Corresponding author. Tel.: +98 21 7724 0288; fax: +98 21 7322 2705.
E-mail address: Sh_afshar@iust.ac.ir (S. Afshar).

to be a mixture of cuprous iodide, molecular iodine and potassium sulfate. The mixture was washed with deionized water to remove molecular iodine and potassium sulfate salt. The precipitate was then dried in a vacuum oven at 100 °C for 2 h to obtain the final grayish white product.

X-ray powder diffraction pattern (XRD) was performed on a SIEFERT XRD 3003 PTS diffractometer using Cu K α irradiation ($\lambda=1.5418$ Å). Scanning electron micrographs and energy dispersive X-ray Spectroscopy were taken with a ZEISS-DSM 960A microscope with attached camera operating at 30 kV to determine the morphology, particle size and high purity of as-prepared synthesized salt. Diffuse reflectance spectrum (DRS) was measured on a TU-1901 spectrophotometer. All the measurements were done at room temperature.

3. Results and discussion

Copper forms a variety of compounds with oxidation states of +1 and +2. In aqueous solution and for the most ligands, it is the +2 oxidation state of the copper that dominates and is the more stable one. However, the reducing ligands, like iodide would reduce the Cu²⁺ ions to the +1 oxidation state. Therefore, after milling the precursors, the brown powder as product was expected to be a mixture of cuprous iodide, molecular iodine and potassium sulfate.

The reaction equation is as follows:



In this reaction, Cu²⁺ and I[−] act as oxidizing and reducing agents, respectively.

To explore the formation of the CuI and in order to identify its phase and structure X-ray diffraction pattern was used. 2θ values and their matching hkl indices are shown in Fig. 1, stating the gamma phase of CuI (JCPDS Card No. 06-0246) and indicating the formation of CuI in a cubic lattice with cell parameter (a) equal to 6.06 Å. This ratify that CuI has been formed without any impurity [15]. The particle size of the CuI powder was estimated by applying Scherrer's equation [20] and was found to be equal to 40 nm.

The morphology of the obtained product was investigated by scanning electron microscopy (SEM). As can be seen in Fig. 2a and b, the SEM images show that the CuI powder is consisted of spherical nanoparticles with the size of 40–70 nm, which is very close to that obtained by XRD analysis. Also the crystallites are uniformly aggregated.

The purity of the prepared CuI nanoparticles was also evidenced by the EDX pattern showed in Fig. 2c. The EDX measurement verified the existence of copper and iodide in the final product. This analysis showed a purity of 100% for the synthesized compound which is in good agreement with the XRD data.

The optical absorption of the as-prepared CuI was investigated in the wavelength range of 300–800 nm. Based on the theory of optical absorption, the relation between the absorption coefficient and photon energy is as follows [20]:

$$ah\nu = A(h\nu - E_g)^{1/2} \quad (2)$$

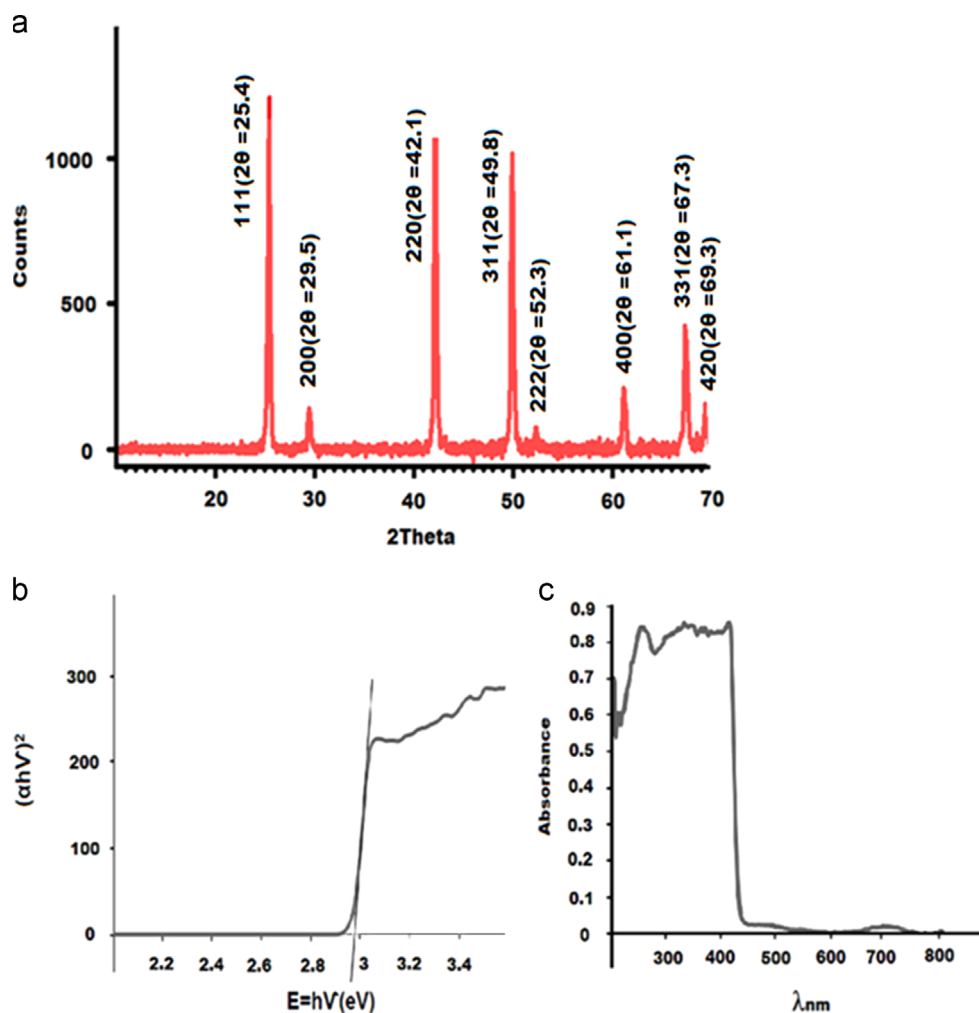


Fig. 1. (a) XRD pattern of the CuI nanoparticles; (b) the plot of $(ah\nu)^2$ vs. $h\nu$ and (c) optical absorption spectrum of obtained CuI nanoparticles.

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