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Research Note

Fabrication of Nanosized Cuprous Oxide Using Fehling's Solution

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Abstract. *In this paper we describe a facile method for the synthesis of Cu_2O nanoparticles by reduction of Fehling's solution, using glucose as reducing agent. Copper sulfate is used as a precursor with potassium sodium tartarate in an alkaline media to produce Fehling's solution. The precipitation of Cu_2O nanoparticles from this solution in the presence of glucose was controlled by addition of SLES or Triton-X 100 as surfactants. The reactions have been carried out at 60° C with high repeatability. The purification process of the Cu_2O product does not require expensive methods, since a solid product is obtained from a reaction in liquid phase. The resulting Cu_2O nanoparticles were characterized by X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Energy-Dispersive X-ray spectroscopy (EDX), Transmission Electron Microscopy (TEM) and Fourier-transform infrared (FTIR) spectroscopy.*

Keywords: *Cu_2O; Fehling's solution; Nanoparticles; Surfactants; SLES; Triton-X 100.*

INTRODUCTION

There has been increasing interest in the synthesis and study of inorganic nanostructures in recent years for their widely varying properties and potential applications [1-4]. Metal oxide nanostructures with well-controllable size and shape have received increasing attention in current material synthesis and devices fabrication [5-7]. Among the various transition metal oxides, cuprous oxide (Cu_2O) is an important p-type and transparent semiconductor. In this oxide, which has a direct band gap of 2.0 eV., the semiconductivity is due to the presence of Cu^+ vacancies or cation-deficiency (about 1.5 to 3%). Cu_2O is currently attracting considerable interest in the fields of both condensed matter physics and material chemistry. This interest is mainly to do with its rich excitonic structure and potential applications in solar energy conversion, catalysis, sensing, magnetic storage and electrode materials in lithium ion batteries etc. [8-12]. Furthermore, the discovery that illuminated Cu_2O could act as a stable photocatalyst, for the photochemical decomposition of water into O_2 and H_2 under visible light irradiation [13] is one of the exciting developments in this field. In addition, Cu_2O is a prospective candidate for a low-cost photovoltaic power generator because of its high optical absorption coefficient and reasonably good photovoltaic properties [14].

Cuprous oxide belongs to the space group $Pn\bar{3}m$, and the unit cell contains two copper and four oxygen ions. These are arranged with oxygen atoms in a body centered cubic lattice surrounded tetrahedrally by copper ions. This arrangement is different from that of CuO in which four oxygen atoms surround the copper ion with square planar geometry. Therefore, these two close copper oxides show quite distinct XRD spectra and differ in stability.

Generally, Cu_2O nanostructures are either gained via oxidation of pure copper [15] or obtained via reduction of Cu^{2+}. In the second method, a certain reducer is additionally introduced to the reaction system to obtain Cu_2O crystals. Up to now, a variety of approaches including electrochemical deposition, catalytic reduction, solvothermal methods, seed-mediated synthesis and microemulsions have been used to prepare nanosized Cu_2O with different morphologies such as cubes, octahedrons, pyramids, cages or hollow spheres [16-21].

It is well known that various surfactants including alkyl amines, alkyl acids, alkylphosphonic acid, trioctylphosphine oxide, cetyltrimethylammonium bro-

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mide (CTAB), Sodium Dodecyl Sulfate (SDS) and Triton-X 100 are frequently used as capping agents to tailor the crystal shape in high-temperature solution phase synthesis [22-29]. Although there are enormous quantity of reports for the synthesis of nanosized Cu_2O with different morphologies, it is important to note that most of these techniques have commonly resulted in a simultaneous growth of CuO and Cu_2O nanocrystals or Cu_2O nanoparticles modified with CuO monolayer shell [30,31]. Some reported procedures for the preparation of nanostructured Cu_2O required the use of either various complex or lengthy and cumbersome materials [32,33]. Hence, the ability to grow high quality nanoparticles is so far limited for Cu_2O, and developing effective and facile methods for the preparation of high-quality Cu_2O nanocrystals is still in progress. In this paper, we demonstrate a facile and reproduceable method for synthesizing high-quality Cu_2O nanoparticles using the familiar Fehling's solution. This solution was easily reduced by glucose in the presence of either Triton-X 100 or SLES (Sodium Laureth Sulfate) surfactants to afford Cu_2O nanocrystals in quantitative yield.

EXPERIMENTAL PROCEDURE

Materials

All the chemicals including $CuSO_4.5H_2O$, potassium sodium tartarate tetrahydrate, Triton-X 100, sodium laureth sulfate and NaOH were of analytical grade and were used as received from either Merck or Fluka without further purification.

Preparation of Cu_2O Nanoparticles

Fehling's solution which is comprised of equal parts of the following solutions was first made:

Solution 1: Was made by dissolving of copper (II) sulfate pentahydrate (6.9 g 0.02 mol) in distilled water (100 mL).

Solution 2: Was made by dissolving of potassium sodium tartarate tetrahydrate (34.6 g) and sodium hydroxide (12 g) in distilled water (100 mL).

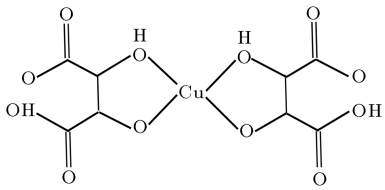
50 mL of each of the above solutions were mixed together in a beaker and Triton X-100 or SLES (2 g) was added to the mixture and vigorously stirred for 15 m. To this mixture, aqueous solution containing 5 g of glucose in 50 mL water was added and the whole content was then heated at 60°under continuous stirring. A brick-red solid of Cu_2O precipitated after a short period of reaction time. The solid was filtered off, washed with deionized water (3 times) and ethanol (2 times) and it was dried in an oven at 80°C for 3 h to afford nanosized Cu_2O, 1.38 g (97% yield based on the used copper sulfate; each mole of copper sulfate produces 0.5 mole of Cu_2O).

Characterization

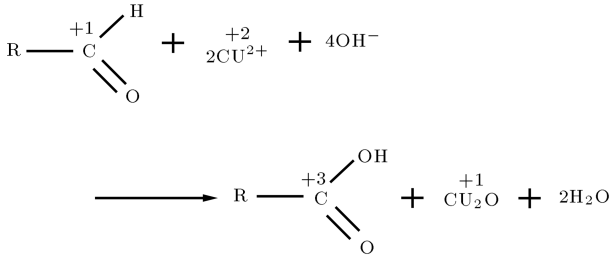
Characterization of the as-prepared Cu_2O nanoparticles was carried out by different techniques. The morphologies and compositions of the Cu_2O nanoparticles were examined by Scanning Electron Microscopy (SEM), using a LEO 1455 VP equipped with an energy-dispersive. X-Ray Diffraction (XRD) patterns were recorded with a Philips analytical X-ray diffractometer using CuKα radiation (λ = 1.54056 Å). The TEM images were taken using a LEO 906 E transmission electron microscope.

RESULTS AND DISCUSSION

Fehling's solution contains tartarate ion in addition to Cu^{2+} and sodium hydroxide. The tartarate ion acts as a complexing agent to keep the copper ion in solution. The coordination mode of the bidentate tartarate ligand to the copper metal is shown below:



Without tartarate ions, cupric hydroxide, $Cu(OH)_2$, would instantly precipitate from the basic solution of Cu^{2+}. However, heating of Fehling's solution in the presence of a mild reducing agent, such as glucose, causes the reduction of Cu^{2+} to Cu^{+}. The tartarate ion is unable to complex cuprous ion Cu^{+}, so the reduction of Cu^{2+} to Cu^{+} by reducing sugars results in the formation of an orange to red precipitate of Cu_2O. In fact, the contact of cupric ion (Cu^{2+}) in the tartarate complex with the aldehyde group of glucose molecule reduces it to a cuprous ion, which then precipitates as red Cu_2O and the aldehyde is oxidized to a carboxylic acid. The chemical reaction which occurs in this process can be shown in the following equation:



In fact, the reduction of Cu^{2+} to Cu^{+} plays the main role first in this process and then Cu_2O is generated through the hydrolysis of Cu^{+} according to the following reactions [31]:

$$Cu^{2+} + H_2O \rightarrow CuOH + H^{+},$$

$$2CuOH \rightarrow Cu_2O + H_2O.$$

Therefore, we took advantage of the ready reactivity of Fehling's solution with glucose to innovate a facile method for the synthesis of Cu_2O nanoparticles using either Triton X-100 or SLES as surfactants to control the size of these particles. Using an excess of glucose will insure the complete conversion of Cu^{2+} to Cu_2O, but no further reduction of this cuprous oxide to copper metal was observed.

The surfactant-assisted method is an effective process to prepare size controllable nanocrystals which is simple, convenient and low cost route. Surfactant molecules may act as a growth controller, as well as an agglomeration inhibitor. This is done by forming a covering film on the newly formed particles to prevent their agglomeration [34].

In our procedure, the used surfactants, i.e. SLES and triton X-100, may act in the same manner to reduce the size of Cu_2O nanoparticles. The procedure of preparing Cu_2O nanoparticles has been repeated several times, and more or less the same product was obtained.

The crystal structure of the Cu_2O product was confirmed by X-ray diffraction. An XRD pattern of the as-prepared nanosized Cu_2O was given in Figure 1. The XRD spectrum contains five peaks that are clearly distinguishable. All of them can be perfectly indexed to crystalline Cu_2O not only in peak position, but also in their relative intensity. The peak positions are in good agreement with those for Cu_2O powder obtained from the International Center of Diffraction Data card (JCPDS file no. 05-0667) confirming the formation of a single cubic phase Cu_2O with a cuprite structure. The peaks with 2θ values of 29.601°, 36.521°, 42.441°, 61.541°, 73.691°and 77.611° correspond to the crystal planes of 110, 111, 200, 220, 311 and 222 of crystalline Cu_2O, respectively. No characteristic peaks of Cu metal or CuO are observed in the XRD patterns, indicating that phase-pure cuprous oxide is readily obtained in the solution phase by reduction of Fehling's solution with glucose. The broadness of the peaks can be used to calculate crystallite size of Cu_2O particles by using Debye-Scherrer formula [35]. The mean size of these particles was estimated to be about 20 and 30 nm for the used surfactant SLES and Triton X-100, respectively. The XRD spectra of both samples of Cu_2O prepared in the presence of Triton X -100 or SLES are more or less the same, therefore only one XRD spectrum (the one which belongs to Triton X -100) is shown in Figure 1.

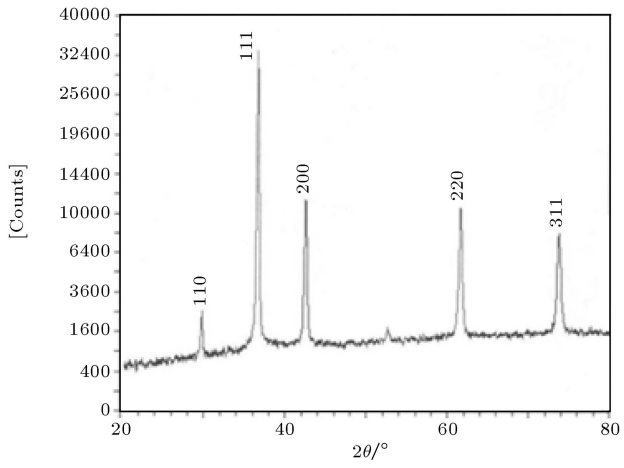


Figure 1. XRD patterns of Cu_2O nanoparticles.

Figure 2 represents the Transmission Electron Microscopy (TEM) images of the obtained Cu_2O. TEM image of the Cu_2O shows well dispersed roughly spherical particles. The mean sizes of Cu_2O particles are about 33 nm and 35 nm for the surfactants SLES and Triton X-100, respectively. The size of Cu_2O nanoparticles obtained from the TEM studies are in close agreement with those calculated from XRD diffraction patterns.

Scanning Electron Microscopy (SEM) was used to further identify the morphology of the as-synthesized Cu_2O. As shown in Figure 3, the prepared cuprous oxide displays a lot of stacked spheres and semispheres with almost uniform diameters. It appears that theses spheres and semispheres have rough surfaces and may be composed of smaller nanoparticles.

EDX analysis was also used to determine the composition of the Cu_2O nanoparticles. As seen in Figure 4, the EDX spectrum of the obtained Cu_2O indicates the existence of only copper and oxygen elements. Cu_2O indicates the existence of only copper and oxygen elements. Furthermore, according to the

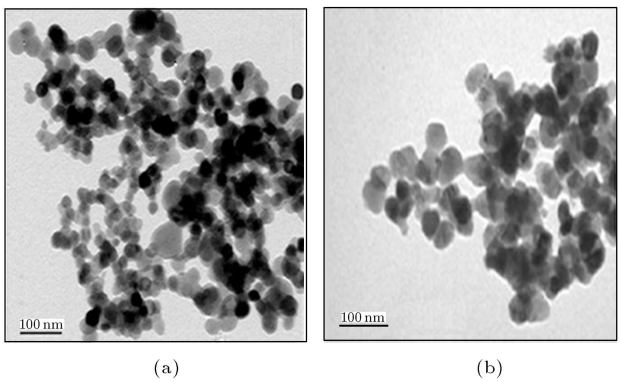
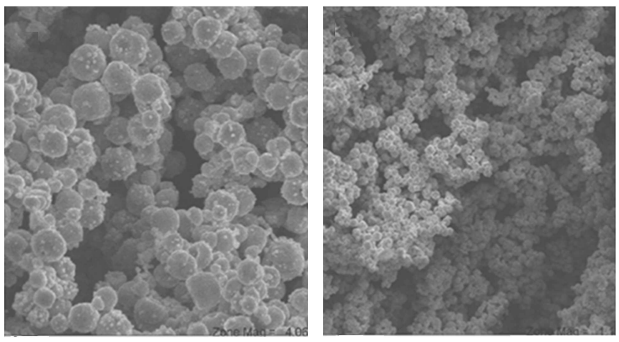


Figure 2. TEM images of Cu_2O nanoparticles obtained in the presence of (a) Triton-X 100 and (b) SLES.



(a) (b)

Figure 3. SEM images of as-prepared Cu_2O in the presence of (a) Triton-X 100 and (b) SLES.

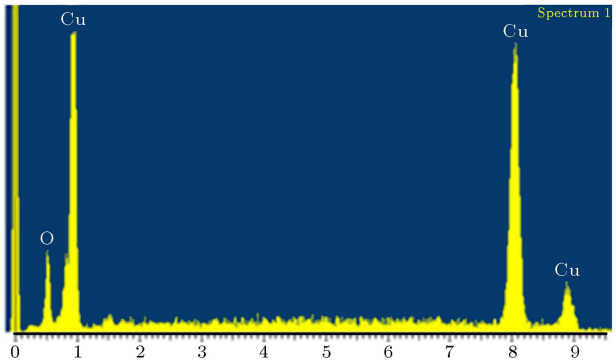


Figure 4. EDX spectrum of Cu_2O nanoparticles.

EDX analysis results (see Table 1), the prepared nanoparticles are almost pure Cu_2O with no Cu or CuO impurities.

As shown in Table 1, the elemental analysis of the as-synthesized cuprous oxide, using either Triton X-100 or SLES surfactants, is in a close consistence with the expected theoretical percentages of Cu and O elements in Cu_2O.

The as-prepared nano cuprous oxide was further characterized by Fourier-transform infrared (FTIR) spectroscopy. This spectroscopy has long been utilized as a powerful tool to provide supplementary information on the nature of copper oxides [36,37]. The FTIR spectrum of the as-synthesized Cu_2O in the wavenumber range 400-1000 cm^{-1}, as shown in Figure 5, displays an absorption peak at around 623 cm^{-1}, which can be attributed to the Cu (I)-O vibration. This observation excludes the presence of any cupric oxide (CuO) impurity, since CuO exhibits three obvious absorption peaks at around 588, 534 and 480 cm^{-1}, which can be assigned to the vibrations of Cu (II)-O bonds. The present FTIR spectrum in Figure 5 is well consistent with that of Cu_2O reported in the previous literatures [38,39].

Table 1. EDX elemental analysis of as-prepared Cu_2O nanoparticles.

Elemental Analysis Calculated: Cu: 88.82 %, O: 11.18 % Experimental		
Sample	**Cu %**	**O %**
Cu_2O (Triton X-100)	88.76	11.24
Cu_2O (SLES)	88.62	11.38

The obvious difference between the FTIR spectra of Cu_2O and CuO can be explained on the bases of their geometry or coordination number and the electronic configurations of the cations. In its crystal lattice, Cu_2O is connected to two oxygen atoms in a linear coordination, whereas CuO is linked with four oxygen atoms in a square planar geometry. Therefore, the Cu(I)-O bonds in Cu_2O are stronger than Cu(II)-O bonds in CuO and this will clearly explain why the vibration of the Cu-O bond in Cu_2O appears in higher frequencies than those of CuO. Moreover, the Cu^+ ion has a symmetrical d^{10} electronic configuration which leads to equivalent Cu-O bonds but this is not the case in CuO. The Cu^{2+} cation in CuO has a d^9 configuration which leads to Jahn-Teller distortion and different Cu-O bonds is expected. Consequently, the vibrations of these nonequivalent bonds in CuO appear in different frequencies; three peaks in the FTIR spectrum instead of one peak for Cu_2O.

CONCLUSION

In summary, Cu_2O nanoparticles with diameters of about 30 nm have been successfully synthesized via reduction of Fehling's solution (an alkaline solution of copper sulfate and tartarate ions) by glucose in the presence of a surfactant. This method employed an inexpensive, easy control, reproducible and simple

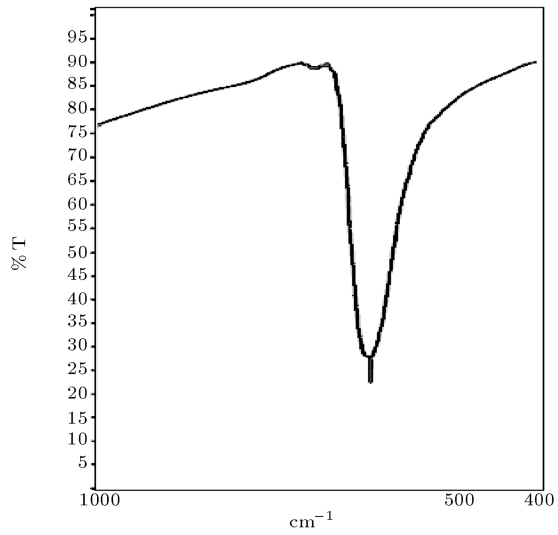


Figure 5. FTIR of as-prepared fabricated Cu_2O.

process for a large-scale synthesis of cuprous oxide nanoparticles. The as-prepared Cu_2O nanoparticles were obtained with high purity and almost quantitative yield. The obtained Cu_2O was characterized by XRD, TEM, SEM, EDX and FTIR techniques.

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BIOGRAPHIES

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Leila Matouri received her B.S. in chemistry in 1997 and her M.S. in Inorganic Chemistry in 2008 from Shahid Chamran University, Ahvaz, Iran. During her M.S. course she has been involved in synthesizing of a variety of nanostructured metal oxides using microwave-assisted reactions and some other procedures. She has had so far six presentations in national conferences and one journal paper published. She is currently teaching chemistry in a high school in Ahvaz.