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Original article

Novel synthesis and characterization of CuO nanomaterials: Biological applications

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ABSTRACT

CuO nanoparticles were synthesized at a relatively low temperature (80 °C) for 2 h using polyethylene glycol–glycerol mixture which acts as a capping agent. A detailed characterization of the synthesized nanomaterials were performed utilizing X-ray diffraction (XRD), infra-red spectroscopy (IR), thermogravimetric analysis (TGA-DTA), transmission electron microscopy (TEM), photoluminescence (PL) by studying its crystalline phase, vibrational mode, thermal analysis, morphology and photoluminescence properties. The effect of annealing on the as-prepared nanoparticles were studied and compared with their corresponding bulk counterpart. The synthesized nanoparticles have been screened for *in vitro* cytotoxicity (IC_{50}) studies against the human cervical adenocarcinoma cell line (HeLa) using MTT assay methods. The as-prepared nanoparticle inhibits the proliferation of this HeLa cell. The standard disc diffusion method has been used to study the antibacterial activity of the samples against the human pathogenic bacteria *Escherichia coli* (MTCC 729), *Proteus mirabilis* (MTCC 425) and *Klebsiella pneumoniae* subsp. *pneumoniae* (MTCC 432). The results have been compared with the positive control antibiotic gentamycin. The synthesized nanoparticles would provide a potential alternative to antibiotics for controlling some of the microorganisms causing urolithiasis.

1. Introduction

Nanotechnology has attracted global attention because materials at nano-scale have properties unique from their bulk equivalents. As the size of materials is reduced to the nanometer regime the resulting properties change noticeably. Considerable efforts have been made to characterize and describe the physical and chemical properties of metal oxide nanomaterials because of their significant applications in numerous technological fields [1,2]. The oxides of transition metals are an important class of semiconductors that have wider applications in magnetic storage media, solar energy transformation, electronics, catalysis, *etc.* [3–6]. Among various transition metal oxides, copper oxide (CuO) has attracted greater attention due to its fascinating properties such as the basis of high critical temperature (T_c) superconductors. CuO is a semiconducting compound with a narrow band gap and is used for photoconductive and photothermal applications [7]. An improved understanding of nanoparticles and biological cell interactions can lead to the development of new sensing, diagnostic, and treatment capabilities, such as improved targeted drug delivery, gene therapy, magnetic resonance imaging (MRI) contrast agents, and biological warfare agent detection [8–10]. Nano-sized metallic copper and its oxides possess good potential for photo-catalytic [11], sensing applications [12] and their usage in bio-related fields including fouling control and nano-toxicology [13] is also being explored.

Nanotoxicology was proposed as a new branch of toxicology to address the gaps in knowledge and to specifically address the adverse health effects likely to be caused by nanomaterials [14]. Nanotoxicology is involved in proposing reliable, robust, and data-assured test protocols for nanomaterials in human and environmental risk assessment [15]. With the increasing demand of nanomaterials in the field of biological applications, cytotoxicity of the nanomaterials becomes a major concern. Copper oxide nanoparticles appeared to have greater cytotoxicity compared to their bulk counterpart and to other metal oxides nanoparticles [16].

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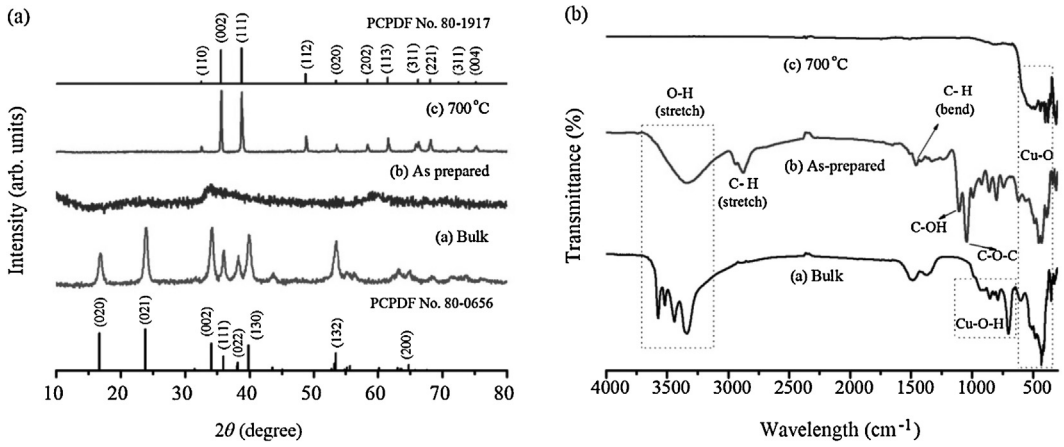

Fig. 1. (a) XRD spectra (along with PCPDF Card No. 80-0656 for $Cu(OH)_2$ and 80-1917 for CuO) and (b) IR spectra of bulk and CuO nanoparticles (as-prepared and 700 °C annealed samples).

Recently, our research group has reported the preparation of nanomaterials using polyethylene glycol (PEG) along with glycerol as the capping agent cum medium [17,18]. The importance of using such long chain hydrocarbon in the synthesis of nanomaterials is the formation of monodispersed nanomaterials. Apart from that, PEG is also considered as one of the best non-ionic polymer used in biomedical science especially in the delivery of anticancer drugs and other bioactives clinically. It is considered as a versatile candidate for the prodrug conjugation due to its high solubility in the aqueous medium. The covalent attachment of PEG (PEGylation) of peptides proteins, drugs, and bioactives is known to enhance the aqueous solubility of hydrophobic drugs, prolong circulation time, minimize nonspecific uptake, and achieve specific tumor targetability through the enhanced permeability and retention effect. Numerous PEG-based therapeutics have been developed, and several have received market approval. A vast amount of clinical experience has been gained which has helped to design PEG prodrug conjugates with improved therapeutic efficacy and reduced systemic toxicity [19].

In the present study, CuO nanomaterials were prepared by a novel synthesis technique using PEG-Glycerol as the capping agent cum medium and their applicability in the field of biological science have been studied. Detail characterization and the effect of annealing on the properties of CuO nanomaterials have also been investigated. The work further investigate how CuO nanoparticles interact with human cervical cancer cell (HeLa) and three human urolithiasis inducing bacteria such as *Escherichia coli* (MTCC 729), *Proteus mirabilis* (MTCC 425) and *Klebsiella pneumoniae* sub sp. *pneumoniae* (MTCC 432).

2. Experimental

CuO nanomaterials were prepared by a relatively novel synthesis route where PEG is used as a capping agent and EG as the solution medium. The prepared nanomaterials were annealed at higher temperature to analyze the effect of annealing temperature on the properties of the synthesized nanomaterials. Detail experimental procedure and analysis about the phase formation of the CuO nanomaterials along with IR studies (Fig. 1) are shown in Supporting information.

3. Results and discussion

3.1. Thermal analysis (TGA-DTA)

The thermal properties of as-prepared $Cu(OH)_2$ nanoparticles were investigated by thermogravimetric analysis (TGA) and differential thermal analysis (DTA), as shown in Fig. 2a. The initial weight loss of ~45% in the TGA curve up to 220 °C, accompanied by an endothermic peak at 170 °C, is assigned to the loss of free and

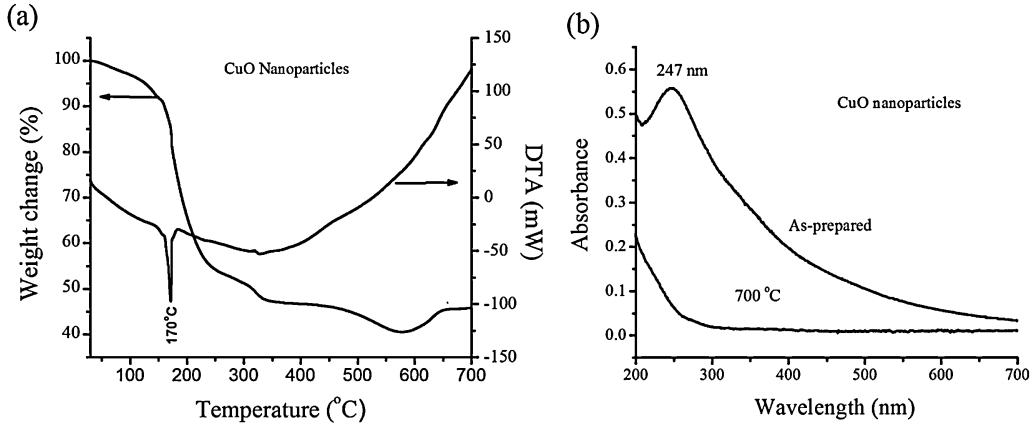

Fig. 2. (a) Thermal analysis (TGA-DTA) along with (b) UV–vis spectra of CuO nanoparticles.