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A facile soft template synthesis and characterization of $PbHAsO_4$ nanocrystals

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Abstract

Monoclinic lead hydrogen arsenate (LHA) nanocrystals with different crystallization morphologies and crystallite sizes were prepared successfully by a soft template synthesis method in the presence of sodium dodecylbenzenesulfonate (SDBS) or polyvinylpyrrolidone (PVP). The products were characterized by X-ray diffraction (XRD) and transmission electron microscopy (TEM). The possible mechanism of SDBS and PVP in the experiment was briefly illustrated.

Keywords: A. Nanostructures; B. Chemical synthesis; C. X-ray diffraction

1. Introduction

Recently, nanostructured materials have drawn much attention because they were expected to play a key role in basic scientific research and fabricating nanodevices with remarkable optical, electric and magnetic properties [1–3]. Various preparation methods towards diverse nanomaterials, including templating direction, chemical vapor deposition, and solution-based solvothermal or hydrothermal treatment have been extensively developed [4–6]. Among these methods, template synthesis has been

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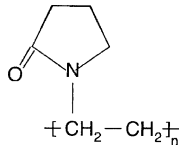

Scheme 1. The structure of PVP molecule.

one of the most popular methods. Various nanomaterials have been prepared by using hard templates such as carbon-nanotube [7] and porous Al_2O_3 [8] and soft templates such as polymer [9] and surfactants [10]. In recent years, a great progress has been made in the effect of anionic surfactant SDBS on crystallization progress and controlled shapes of inorganic materials [11,12]. The surfactant not only provides favorable site for the growth of the particulate assemblies, but also influences the formation progress, including nucleation, growth, coagulation and flocculation. PVP (Scheme 1) a water soluble polymer has an amphiphloic character due to its unique structure consisting of a polar imide group and non-polar methylene and methane groups [13]. It has shown to exhibit well compatibility with many inorganic materials and been widely used to prepare nanomaterials [14,15].

LHA is well known as an important hydrogen-bonded ferroelectric with a second-order transition from a monoclinic *Pc* to a paraelectric *P*2/*c* at *Tc* = 312 K, which is usually grown from the gel-growth method with large sizes [16]. As far as we know, the synthesis of LHA nanocrystals has not been reported in any literature up to now. To obtain new types of applications or enhance the existing devices as a result of size confinement, we have successfully prepared the LHA nanocrystals by the soft template synthesis method in the presence of SDBS or PVP.

2. Experimental procedure

Lead acetate trihydrate ($Pb(CH_3COO)_2{\cdot}3H_2O$ (A.R.)), sodium dodecylbenzenesulfonate ($C_{18}H_{29}NaO_3S$ (A.R.)), polyvinylpyrrolidone (molecule weight: ∼30,000 g/mol (A.R.)), sodium hydroxide (NaOH, 1 M) solution, arsenate acid (H_3AsO_4, 85%) solution were used as starting materials. First, 11.4 g $Pb(CH_3COO)_2{\cdot}3H_2O$ and 2 g SDBS (or 1.5 g PVP) were dissolved in 50 ml de-ionized water, respectively, and then the two solutions were mixed completely under magnetic stirring for half an hour. Then 2.4 ml 85% H_3AsO_4 solution was added dropwise (about 1 drop/s) to the mixture and stirred for 4 h. At the same time the PH value was adjusted to be about 5 by the addition of 1 M NaOH solution. Finally, the mixture was filtered, and the obtained white precipitation was washed several times with de-ionized water and absolute ethanol and subsequently dried in an oven at 90 °C for 12 h.

The X-ray diffraction (XRD) pattern of the powder was examined using Japan RigaKu *D*/max-γ X-ray diffractometer system with graphite monochromatized Cu Kα irradiation (λ =1.5418 Å). The transmission electron microscopy (TEM) was taken using a JEM-100CX transmission electron microscopy running at an accelerating voltage of 100 kV. The excitation and emission spectra of the sample were measured by an Edinburgh FLS920 fluorescence-lifetime and steady-state spectrometer. All the measurements were taken at room temperature.