Synthesis and characterization of nanowires Hausmannite (Mn$_3$O$_4$) by solid-state thermal decomposition

ABSTRACT

In this study, we synthesis one-dimensional (1D) manganese(III) Schiff base coordination polymer [Mn(Brsalophen)(μ$_1$,3-$N_3$)$_n$] by reaction of MnCl$_2$·6H$_2$O and tetradeutate Schiff base ligand Brsalophen at the presence of NaN$_3$ in methanol and characterized by elemental analyses (CHN) and FT-IR spectroscopy. It was used as a new precursor to prepare spinel type manganese oxide nanowires by a facile solid-state thermal decomposition in air at 400ºC for 3.5 h. The crystallinity, purity and morphology of the Mn$_3$O$_4$ products were characterized by powder X-ray diffraction (XRD) and scanning electron microscopy (SEM). The powder X-ray diffraction and scanning electron microscopy confirmed that the prepared Mn$_3$O$_4$ nanowires are pure single phases. The present method allows preparation of the Mn$_3$O$_4$ nanowires without expensive or toxic organic solvent and complicated equipment. The nanowires have a diameter about ≈25 nm and length exceeding 1.5 μm. It has potential to be applied as a general method for preparation of other transition metal oxide nanoparticles.

Keywords: Manganese(III); Nanowires; Thermal decomposition; XRD; SEM.

INTRODUCTION

In recent years, the synthesis of uniformly sized and shape-controlled transition metal oxides has attracted a growing interest. The interest originated not only in novel chemical and physical properties [1,2] of the nanoparticles, but also in many technological applications, in e.g. catalysis, sensing and magnetic resonance imaging [3-5]. Mn$_3$O$_4$ is one of the most stable oxides of manganese and if found wide range of applications in exchange, molecular adsorption, electrochemistry and solar energy transformation [6-9]. Until now, various nanostructures of Mn$_3$O$_4$, such as nanoparticles, nanorods and other structures [10-12], have been synthesized by different methods [13-16]. Among these methods the thermal decomposition distinguishes by its simplicity, reproducibility and low costs [17,18].
Salavati-Niasari et al. [17,18] prepared Mn$_3$O$_4$ nanoparticles by thermal decomposition of Mn(sal)$_2$ and Mn(HNA)$_2$ in oleyl amine; Morsali et al. [19-22] used for the same purpose various polymeric coordination compounds. Herein, we report on the synthesis of Mn$_3$O$_4$ nanowires from the thermal decomposition of 1D manganese(III) coordination polymer [Mn(Brsalophen)(μ$_{1,3}$-N$_3$)]$_n$. To the best of our knowledge, this is the first report on the preparation of Mn$_3$O$_4$ from this precursor.

EXPERIMENTAL

Materials and characterization

All reagents and solvents for synthesis and analysis were commercially available and used as received without further purifications. X-ray powder diffraction (XRD) pattern of the nanowires was recorded on a Bruker AXS diffractometer D8 ADVANCE with Cu-Kα radiation with nickel beta filter in the range 2θ = 10°–90°. Scanning electron microscopy (SEM) images were obtained on Philips XL-30ESEM.

Preparation of Mn$_3$O$_4$ nanowires

The precursor complex [Mn(Brsalophen)(μ$_{1,3}$-N$_3$)]$_n$ was prepared according to the procedure described previously [23,24]. Then, the precursor complex was loaded to a platinum crucible placed in an oven and heated at a rate of 10°C/min in air. Nanoparticles of manganese oxide were synthesized at 400°C after 3.5 h. The final products was washed with ethanol for at least three times to remove impurities, if any, and dried at r.t. for 3 days. The obtained Mn$_3$O$_4$ nanowires were characterized by XRD and SEM.

RESULTS AND DISCUSSION

Figure 1 shows the XRD of manganese oxide nanowires. The powders exhibited the crystalline pattern corresponding to the standard Mn$_3$O$_4$ XRD pattern (JCPDS: 24-0734) with all diffraction peaks indexed as a tetragonal phase. No obvious peaks of impurities were found. Moreover, the observed peaks were sharp intense, indicating the well crystallized form of the prepared Mn$_3$O$_4$ nanowires.

The morphology of the Mn$_3$O$_4$ nanoparticles was investigated by scanning electron microscopy (SEM) (Figure 2). The SEM image of the as-prepared Mn$_3$O$_4$ nanowires indicates the uniform size, shape and high purity of the products. The nanowires have a diameter about ≈25 nm and length exceeding 1.5 μm.

CONCLUSIONS

In summary, we have successfully prepared Mn$_3$O$_4$ nanowires by solid-state thermal decomposition of 1D manganese(III) complex [Mn(Brsalophen)(μ$_{1,3}$-N$_3$)]$_n$. This method is facile, inexpensive, and nontoxic can be extended for preparation of other transition metal oxide nanoparticles.
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REFERENCES


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