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Synthesis and characterization of nanowires Hausmannite (Mn_3O_4) by solid-state thermal decomposition

A. A. Dehno Khalaji*
F. Malekan

Department of Chemistry, Faculty of Science, Golestan University, Gorgan, Iran.

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* Corresponding author:
Aliakbar Dehno Khalaji
Department of Chemistry, Faculty of Science, Golestan University, Gorgan, Iran.
Tel+98171 2254882
Fax +98171 2254882
Email alidkhalaji@yahoo.com

ABSTRACT

In this study, we synthesis one-dimensional (1D) manganese(III) Schiff base coordination polymer $[Mn(Brsalophen)(\mu_{1,3}\text{-}N_3)]_n$ by reaction of $MnCl_2{\cdot}6H_2O$ and tetradentate 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_3O_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_3O_4 nanowires are pure single phases. The present method allows preparation of the Mn_3O_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, sensoring and magnetic resonance imaging [3-5]. Mn_3O_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_3O_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_3O_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_3O_4nanowires from the thermal decomposition of 1D manganese(III) coordination polymer $[Mn(Brsalophen)(\mu_{1,3}\text{-}N_3)]_n$. To the best of our knowledge, this is the first report on the preparation of Mn_3O_4 from this precursor.

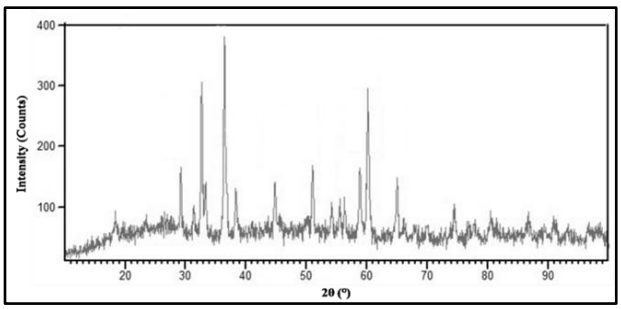

Fig. 1. X-ray diffraction pattern of Mn_3O_4 nanowires.

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 $2\theta = 10^o–90^o$. Scanning electron microscopy (SEM) images were obtained on Philips XL-30ESEM.

Preparation of Mn_3O_4 nanowires

The precursor complex $[Mn(Brsalophen)(\mu_{1,3}\text{-}N_3)]_n$ 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 10ºC/min in air. Nanoparticles of manganese oxide were synthesized at 400ºC 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_3O_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_3O_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_3O_4 nanowires.

The morphology of the Mn_3O_4 nanoparticles was investigated by scanning electron microscopy (SEM) (Figure 2). The SEM image of the as-prepared Mn_3O_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.

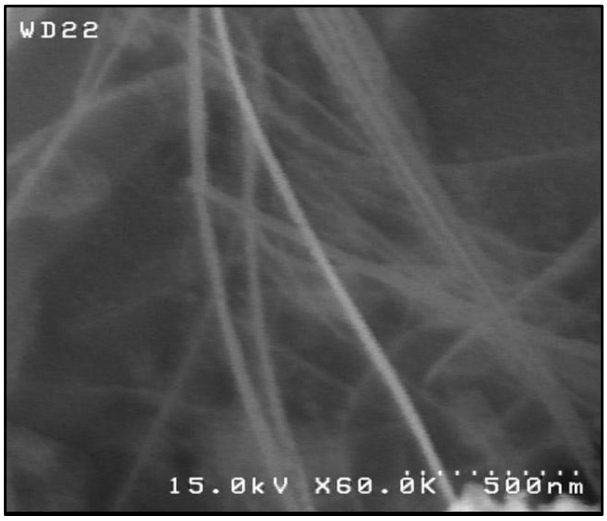

Fig. 2. SEM image of Mn_3O_4 nanowires.

CONCLUSIONS

In summary, we have successfully prepared Mn_3O_4 nanowires by solid-state thermal decomposition of 1D manganese(III) complex $[Mn(Brsalophen)(\mu_{1,3}\text{-}N_3)]_n$. This method is facile, inexpensive, and nontoxic can be extended for preparation of other transition metal oxide nanoparticles.

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