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Cobalt oxide nanoparticles by solid-state thermal decomposition: Synthesis and characterization

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Abstract

In this study, mononuclear octahedral cobalt(III) Schiff base complex [CoL_3], L = (5-bromo-2-hydroxybenzyl-2-furylmethyl)imine was synthesized from the reaction of $Co(NO_3)_2 \cdot 6H_2O$ and the Schiff base ligand L in methanol as solvent and characterized by elemental analyes (CHN) and FT-IR spectroscopy. It was used as a new precursor to prepare spinel type cobalt oxide nanoparticles by a facile solid-state thermal decomposition. Controlling the temperature and time, Co_3O_4 nanoparticles were obtained in air at 550 ºC within 3.5 h. The Co_3O_4 nanoparticles were characterized by powder X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The results confirm that the resulting cobalt oxide were prepared during pure single-phases. Using the present method, Co_3O_4 nanoparticles can be produced without using expensive organic solvent and complicated equipment. TEM result showed that the products are almost flat with the size of about 10-50 nm. It has potential to be applied as a general method for preparation of other transition metal oxide nanoparticles.

Keywords: Nanoparticles; Schiff base complex; cobalt oxide; thermal decomposition.

Introduction

The development of transition metal oxide nanoparticles has received considerable interest because of their interesting size-dependent physical and chemical properties and broad application in several important technologies [1,2]. Among these oxides, spinel types, such as Co_3O_4 and Mn_3O_4, have been the subject of scientific and technologie attention owing to their wide range of applications [3,4]. Cobalt oxide is formed in five different oxidation states [5] among which Co_3O_4 and CoO are the most stable and magnetic of them and have been studied by Zhu and co-workers [6]. Until now, different nanostructures of Co_3O_4, including nanotube, nanoplates, nanowalls, nanospheres and etc have been prepared by different methods [7-10]. In order to prepare Co_3O_4, various physical and chemical methods such as sol-gel [1], combustion [2], ultrasound-assisted [11], co-precipitation [12], ball milling [13], and thermal decomposition [5] have been extensively studied. However, most of these methods are toxic and/or expensive. Among various techniques for synthesis of transition metal oxide nanoparticles [2,14-13],

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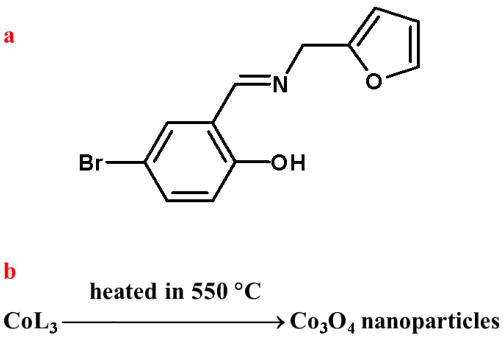
thermal decomposition is one of the best method [14-16], because of its cheapness and non-toxiciy. In addition, the process conditions, particle size, particle crystal structure, and purity could be controlled.

Recently Co_3O_4 nanoparticles were synthesized using thermal decomposition of Co(III) complexes by Farhadi and his co-workers [15,16]. They reported that octahedral cobalt(III) complex was first formed then it was calcined at various temperatures in an electric furnace for 1 h to get cobalt oxide nanoparticles with size about 10-15 nm. Salavati-Niasari and his co-worker synthesized Co_3O_4 nanoparticles by solid state thermal decomposition of tetrahedral cobalt(II) complex $[Co(sal)_2]$ at 500 ºC in an electric furnace for 5 h to get cobalt oxide nanoparticles with size about 20-30 nm [14].

In this paper, we decided to prepare Co_3O_4 nanoparticles from cobalt(III) Schiff base complex $[CoL_3]$ [17] (Scheme 1). The product was identified by powder X-ray diffraction (XRD), Fourier-transformed infrared spectroscopy (FT-IR) and scanning electron microscopy (SEM).



Scheme 1. a) Chemical structure of Schiff base ligand (5-bromo-2-hydroxybenzyl-2-furylmethyl)imine, b) preparation of Co_3O_4 nanoparticles

Experimental

Materials and physical measurements

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 nanoparticles were recorded on a Bruker AXS diffractometer D8 ADVANCE with Cu-Kα radiation with nickel beta filter in the range $2\theta = 4^o–90^o$. Scanning electron microscopy (SEM) images were obtained on Philips XL-30ESEM. Transmission electron microscopy (TEM) images were obtained on a Zeiss EM10C transmission electron microscope with an accelerating voltage of 80 kV.

Synthesis of cobalt complex

The cobalt complex was prepared according to the procedure described previously [17]. $CoCl_2{\cdot}6H_2O$ (1 mmol) was dissolved in 20 mL methanol and stirred for 10 min. About 10 mL methanolic solution of the Schiff base (5-bromo-2-hydroxybenzyl-2-furylmethyl)imine (3 mmol), was added to it dropwise.the mixture was allowed to stir for 2 h at 50 °C. The precipitates of the complex were filtration. *Anal calc*. for $C_{33}H_{27}N_3CoBr_3O_6$: C, 46.07; H, 3.14; N, 4.88%. Found: C, 46.15; H, 3.19; N, 4.93%. FT-IR (cm^{-1}): 1612 cm (C=N).

Synthesis of Co_3O_4 nanoparticles

The precursor complex (0.5 g) was loaded in to a platinum crucible and then was placed in oven and heated at 550 °C with a rate of 10ºC/min in air. Nanoparticles of cobalt oxide were synthesized after 3.5 h (about 0.07 g). The final products were washed with ethanol for at least three times to remove impurities, if any, and dried at r.t. The synthesized Co_3O_4 nanoparticles were characterized by XRD, SEM and TEM techniques.

Results and discussion

Figure 1 shows the XRD pattern ($10 < 2\theta < 80$) of the Co_3O_4 nanoparticles. The diffraction peaks at 2θ = 19 (111), 31 (220), 37 (311), 39 (222), 45 (400), 56 (422), 59 (511) and 66 (440), can be indexed to pure Co_3O_4 cubic phase [14,15]. The crystallite size (D_c) is calculated using the Debye-Scherre formula (Eq. 1) from the major diffraction peak of the Co_3O_4 nanoparticles.

$$D_c = 0.89\,\lambda / \beta\cos\theta \qquad (1)$$

Where λ is the X-ray wavelength used in XRD (here, 1.5418 Å), β is the pure diffraction broadening of a peak at half-height and θ is the Bragg angle. Thus, the average diameter of the Co_3O_4 nanoparticles is found as 10-50 nm.

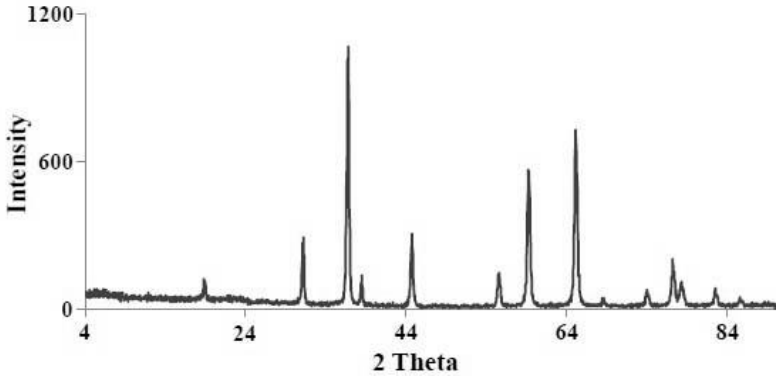


Figure 1. XRD patterns of Co_3O_4 nanoparticles obtained from [CoL_3]

The morphology and microstructure of the Co_3O_4 nanoparticles are investigated by SEM and TEM (Figures 2 and 3). Studies show the particle size of Co_3O_4 nanoparticles is about 10-50 nm. These results indicate that the Co_3O_4 crystals are formed by partially aggregation of smaller crystallites during the synthesis process.

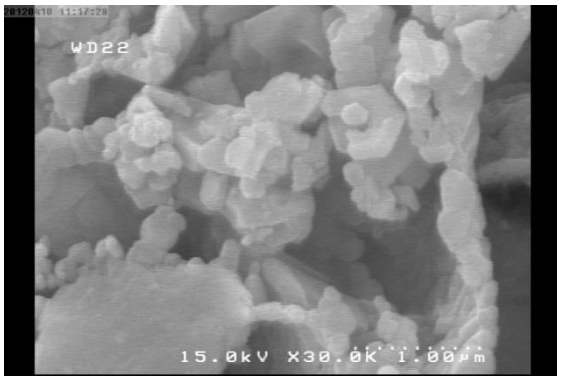


Figure 2. SEM image of Co_3O_4 nanoparticles

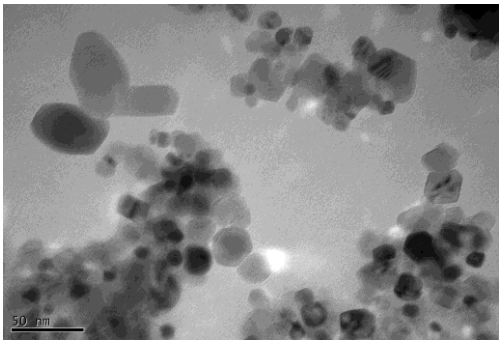


Figure 3. TEM image of Co_3O_4 nanoparticles

Conclusion

Pure Co_3O_4 nanoparticles have been successfully prepared by heating of octahedral cobalt(III) Schiff base complex [CoL_3] at 550ºC. To the best of our knowledge, the synthesis of Co_3O_4 nanoparticles from Co(III) Schiff base complexes has been rarely reported. The method is simple, inexpensive, non-toxic and could be easily extended to other transition metals.

Acknowledgements

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