



Synthesis and characterization of manganese ferrite nanoparticles by thermal treatment method

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ABSTRACT

Cubic structured manganese ferrite nanoparticles were synthesized by a thermal treatment method followed by calcination at various temperatures from 723 to 873 K. In this investigation, we used polyvinyl pyrrolidone (PVP) as a capping agent to control the agglomeration of the nanoparticles. The characterization studies were conducted by X-ray diffraction (XRD) and transmission electron microscopy (TEM). The average particle sizes of manganese ferrite nanoparticles were determined by TEM, which increased with the calcination temperature from 12 to 22 nm and they had good agreement with XRD results. Fourier transform infrared spectroscopy confirmed the presence of metal oxide bands at all temperatures and the absence of organic bands at 873 K. Magnetic properties were demonstrated by a vibrating sample magnetometer, which showed a super-paramagnetic behavior for all samples and also saturation magnetization (M_s) increases from 3.06 to 15.78 emu/g by increasing the calcination temperature. The magnetic properties were also confirmed by the use of electron paramagnetic resonance spectroscopy, which revealed the existence of unpaired electrons and also measured peak-to-peak line width, resonant magnetic field and the g -factor.

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1. Introduction

Recently, many studies have focused on the synthesis of nanomaterials, such as spinel ferrite nanocrystals, which have attracted much attention because of their surface effect (large surface-to-volume ratio) and quantum confinement effects (size-dependent properties). These factors affect their physical and chemical properties. Magnetite and spinel ferrite nanocrystals are regarded as two of the most important inorganic nanomaterials because of their electronic, optical, electrical, magnetic, and catalytic properties, all of which are different from the properties of their bulk counterparts. Spinel ferrites have the structure AB_2O_4 in which A and B display tetrahedral and octahedral cation sites, respectively, and O indicates the oxygen anion site [1]. Among spinel ferrites, manganese ferrite ($MnFe_2O_4$) nanoparticles are very important because they have proven to be useful in many magnetic applications, such as recording media devices [2], drug delivery [3], ferrofluid [4], biosensors [5], and contrast-enhancement agents for MRI technology [6]. The properties of manganese

ferrite nanoparticles are dependent on their micro-structural characteristics, i.e., particle size and shape, which can be controlled in the fabrication processes. In order to achieve materials of the desired physical and chemical properties, the preparation of manganese ferrite nanocrystals through different routes has become an essential part of related research and development activities. Various fabrication methods to prepare spinel manganese ferrite nanocrystals have been reported, including the sol-gel method [7], co-precipitation [8], reverse micelles [9], and the hydrothermal method [10]. Various precipitation agents have been used to produce manganese ferrite nanocrystals of a specific size and shape, e.g., metal hydroxide in the co-precipitation method, surfactant and ammonia in the reverse micelles and micro-emulsion methods, organic matrices in the sol-gel method, and high temperature and pressure in the hydrothermal method. Most of these methods have achieved particles of the required size and shape, but they are impractical for large-scale applications because of they require expensive and complicated procedures, high reaction temperatures, long reaction times, and toxic reagents. They also produce toxic by-products that may harm the environment. In the present study, manganese ferrite nanocrystals were prepared using a thermal treatment method in an aqueous solution containing metal nitrates, polyvinyl pyrrolidone, and deionized water, followed by grinding and calcination.

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No other chemicals were added to the solution. This method offers the advantages of simplicity, low cost, and low reaction temperatures; in addition, it is environment-friendly as it produces no by-product effluents. It must be mentioned that the main drawbacks of this method are removal of PVP and simultaneously controlling particle size of ferrite, moreover it is still limited to some of the materials. The textural and morphological characteristics of the manganese ferrite nanocrystals were studied with various techniques to verify the influence of calcination temperature on the crystallization, morphology, and particle size distribution of the nanocrystals, as well as to explore other parameters of interest.

2. Experimental

Metal nitrate reagents were used as precursors, polyvinyl pyrrolidone (PVP) was used as the capping agent, and deionized water was used as the solvent. Iron nitrate, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, and manganese nitrate, $\text{Mn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, were purchased from Acros Organics with purities exceeding 99%. PVP (MW=29,000) was purchased from Sigma Aldrich and was used without further purification. An aqueous solution of PVP was prepared by dissolving the polymer in 100 ml of deionized water at 343 K; then, 0.2 mmol of iron nitrate and 0.1 mmol of manganese nitrate (Fe:Mn=2:1) were placed in the polymer solution, which was stirred for 2 h using a magnetic stirrer. At the end of the 2 h period, a colorless, transparent solution was obtained. Using a glass electrode, the pH of the solution was determined to be in the range 1–2. No precipitation of materials was observed before the heat treatment. The solution was poured into a glass Petri dish and heated in an oven at 353 K for 24 h to evaporate the water. The dried, orange, solid manganese ferrite that remained was crushed and ground in a mortar to form powder. The calcinations of the powder were conducted at 723, 773, 823, and 873 K for 3 h to decompose the organic compounds and crystallize the nanocrystals.

3. Characterization

The structure of the MnFe_2O_4 nanoparticles was characterized by the XRD technique, using a Shimadzu diffract meter model XRD 6000 employing $\text{Cu K}\alpha$ (0.154 nm) radiation to generate diffraction patterns from powder crystalline samples at an ambient temperature in 2θ range of 10° – 80° . The microstructure and particle size of the nanocrystals were determined from Transmission Electron Microscopy (TEM) images obtained using a JEOL 2010F UHR version electron microscope at an accelerating voltage of 200 kV. FT-IR spectra were recorded using a PerkinElmer FT-IR spectrometer, model 1650. Before recording spectra, the samples were placed on a Universal ATR Sampling Accessory (diamond coated with CsI) and pressed, and then the spectra were recorded. Magnetization measurements were conducted using a vibrating sample magnetometer (VSM) (Lake Shore 4700) at room temperature with a maximum magnetic field of 15 kOe. Electron paramagnetic resonance (EPR) spectra were recorded on a JEOL JES-FA200 EPR spectrometer (JEOL, Tokyo, Japan) at room temperature.

4. Results and discussion

The interactions between PVP and metal ions are represented schematically in Fig. 1, which shows that the manganese (II) and iron (III) ions are bound by strong ionic bonds between the metallic ions and the amide group in a polymeric chain or between the polymeric chains. This uniform immobilization of

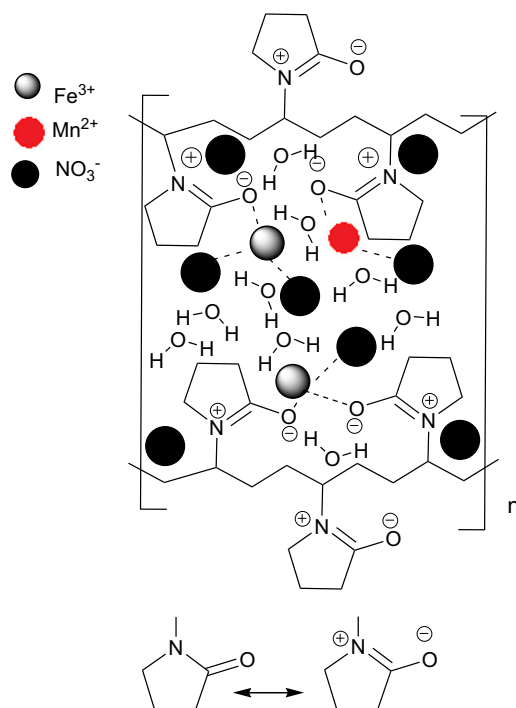


Fig. 1. A proposed mechanism of interactions between PVP and metal ions.

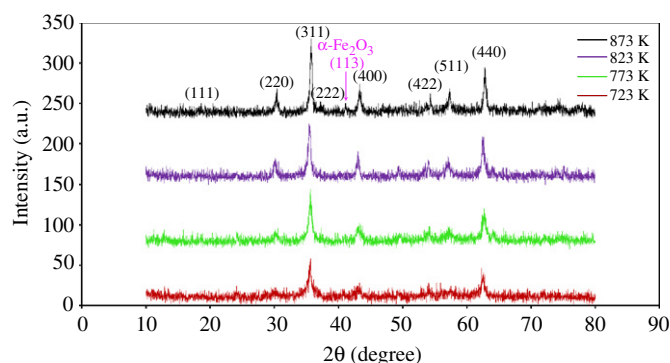


Fig. 2. XRD patterns of manganese ferrite nanoparticles calcined at different temperatures. (For interpretation of the references to color in this figure, the reader is referred to the web version of this article.)

metallic ions in the cavities of the polymer chains favors the formation of a uniformly-distributed, solid solution of the metallic oxides in the calcination process.

The XRD diffraction patterns of the manganese ferrite nanoparticles (JCPDS, 73-1964) are shown in Fig. 2. The patterns show the reflection planes (1 1 1), (2 2 0), (3 1 1), (2 2 2), (4 0 0), (4 2 2), (5 1 1), (4 4 0), and (5 3 1), which confirm the presence of single-phase MnFe_2O_4 with a face-centered cubic structure [11]. The appearance of the plane (1 1 3) is due to the formation of the $\alpha\text{-Fe}_2\text{O}_3$ phase at 873 K (as shown with pink color in Fig. 2), which is evidence of the transfer of Fe^{3+} ions from B site to A site in the mixed spinel structure of MnFe_2O_4 nanoparticles. A neutron diffraction experiment performed on the mixed spinel structure of MnFe_2O_4 nanoparticles showed the existence of Mn^{2+} and Fe^{3+} ions in the sub-lattices of A and B [9].

The average particle size was determined from the full width at half maximum (FWHM) using the well-known Debye–Scherrer equation

$$D = 0.9\lambda / \beta \cos \theta$$

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