Journal of Alloys and Compounds 601 (2014) 116–119

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: www.elsevier.com/locate/jalcom

Synthesis of super paramagnetic particles of $Mn_{1-x}Mg_xFe_2O_4$ ferrites for hyperthermia applications

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ARTICLE INFO

Article history:
Received 20 June 2013
Received in revised form 27 December 2013
Accepted 22 February 2014
Available online 4 March 2014

Keywords:
Spinel ferrites
Nano-particles
Co-precipitation
Hyperthermia

ABSTRACT

A series of $Mn_{1-x}Mg_xFe_2O_4$ (x = 0.0–1.0) spinel ferrites were synthesized by chemical co-precipitation. The materials were investigated by X-ray diffraction, transmission electron microscopy, energy dispersive X-ray spectroscopy and vibrating sample magnetometry. X-ray diffraction patterns for all the samples revealed single phase formation with particle size below 100 nm. The lattice constant was observed to decrease as the Mg-substitution increases thus altering the unit cell volume. Transmission electron micrographs exhibit that the particles are spherically shaped and agglomerated with particle size ranging 52–100 nm quite consistent with particle size obtained from XRD data. The M–H loops for all the samples are narrow with low values of coercivity and retentivity, indicate the super paramagnetic nature of these samples. Based on these characterizations of the samples it is suggested that the $Mn_{1-x}Mg_xFe_2O_4$ ferrites may be potential candidates for hyperthermia applications.

1. Introduction

Hyperthermia has attracted a lot of attention in the recent years. The idea that a localized rise in the temperature can be used to destroy malignant tissues selectively is called hyperthermia. The magnetic powder is injected into the body of the patient and heated to produce local temperature of 43 °C by the application of ac magnetic field. The mechanism of heating for ferromagnetic material critically depends on hysteresis loss. In the case of super paramagnetic particles, heating can occur by the rotation of particles themselves or by the rotation of atomic magnetic moments [1]. The challenging work is the development of magnetic particles with high specific absorption rate (SAR), which allows reduction of ferrofluid dose in vivo. SAR depends on several parameters such that particle magnetization, size and distribution, ac magnetic field and frequency [2]. By controlling the particle size of magnetic particles, one can adjust the heat generation under an oscillating magnetic field [3]. Mn–Zn ferrites are potential magnetic materials high initial permeability, low losses, high saturation magnetization and relatively high Curie temperature having wide applications like soft magnetic powders, hyperthermia, magnetic fluids, heat transfer systems [4,5], transformer core, high velocity magnetic record, resonance magnetic imaging [6] and as magnetoelectric composites [7,8]. For the applications of MFe_2O_4 (where M^{2+} = Mn^{2+}, Zn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, etc.) for future magnetic micro devices to integrate drug delivery systems, a compromise between magnetic moment and absence of magnetic remanent memory is desired, like in super paramagnetic particles [9]. Also the use of magnetostrictive ferrite phase is important and promising to trigger on magnetic-mechanically stimulated drug delivery systems [10]. The possibility of preparing ferrites in the form of nano-particles has open a new and exciting research field with revolutionary applications not only in the electronic technology but also in the field of biotechnology. A remarkable characteristic of spinel structure is that the composition of a given ferrites can be strongly modified while the basic crystalline structure remain the same. Synthesis parameters, composition and microstructure have strong influence on the properties of ferrites. Mix ferrites are very important from technological point of view and substitution of metal cations in ferrites is optimized for having a specific property in the final substituted ferrite for particular application.

The crystallographic, electrical and magnetic properties of these ferrites significantly depend on their method of synthesis, chemical composition, sintering or annealing temperature, substitution of cations and grain size, etc. [11–13]. These parameters control the microstructure forming of the high resistive boundaries between the constituent grains. Properties of ferrites have also been

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http://dx.doi.org/10.1016/j.jallcom.2014.02.138
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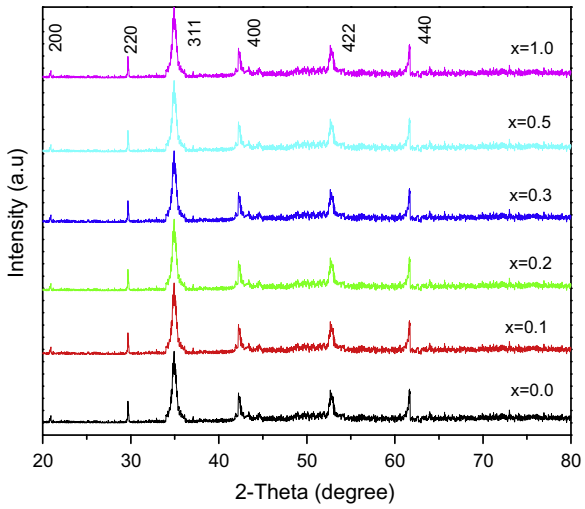

Fig. 1. XRD patterns for all $Mn_{1-x}Mg_xFe_2O_4$ ferrites (x = 0.0, 0.1, 0.2, 0.3, 0.5, 1.0).

Table 1
Lattice constant (a), particle size (D_{xrd}), unit cell volume (V), squareness ratio (M_r/M_s) for all $Mn_{1-x}Mg_xFe_2O_4$ ferrites (x = 0.0, 0.1, 0.2, 0.3, 0.5, 1.0).

Samples $Mn_{1-x}Mg_xFe_2O_4$	a (Å)	D_{xrd} (nm)	V (Å^3)	M_r/M_s
x = 0.0	8.500	72.3	614.13	0.15
x = 0.1	8.490	50.2	611.96	0.12
x = 0.2	8.485	92.4	610.88	0.07
x = 0.3	8.480	79.2	609.80	0.08
x = 0.5	8.475	55.4	608.72	0.08
x = 1.0	8.470	79.2	607.65	0.12

strongly exaggerated when the particle size approaches a critical diameter, below which each particle is considered to be a single domain [14]. Recently, several methods have been used for the synthesis of highly crystalline and uniformly sized magnetic particles of ferrites [15–17]. For the preparation of ferrofluid and magnetic particles with excellent chemical homogeneity, the chemical co-precipitation technique is widely used. This method has gained scientific and technological importance during the last decades. This process offers many advantages as compared to the conventional ceramic method, such as low temperature processing and/or better homogeneity for the synthesis of multi-component materials and thus formation of the homogenized particles of ferrites. The objective of this study is to synthesize and investigate the structural and magnetic properties of Mn–Mg ferrites particles suitable for hyperthermia applications.

2. Experimental

2.1. Synthesis

The Mg-substituted $Mn_{1-x}Mg_xFe_2O_4$ ferrites were prepared by co-precipitation. Analytical grade manganese chloride ($MnCl_2{\cdot}4H_2O$), magnesium oxide (MgO) and iron chloride ($FeCl_3$) were used as starting materials to obtain Fe^{3+}, Mn^{2+}, Mg^{2+} ions in aqueous solutions. After stoichiometric calculations the required amount of parent materials was dissolved in deionized water. The solutions containing these ions were mixed in an appropriate molar proportion in 1000 ml beaker. The solution was heated to 60 °C with continuous stirring and the solution of NaOH and Na_2CO_3 (with ratio 1:4) was used in order to maintain the pH(11–12) value of the solution. The solution of NaOH and Na_2CO_3 was added slowly drop-wise into reactant solution with constant stirring until precipitation is completed. The precipitates were washed off by deionized water for many times. Samples were dried in an oven for 24 hr at 100 °C. The dried powder was sintered at 1000 °C for 6 h followed by the furnace cooling.

2.2. Characterization

The crystal structure of the prepared ferrite powders was identified by Schimadzu X-ray diffractometer (XD5A) using Cu Kα (λ = 1.5406 Å) radiations at room temperature. The surface morphology and microstructure of the samples were studied using a transmission electron microscope TEM (JEOL model JEM1010). The elemental composition was determined by energy dispersive X-ray spectroscopy (EDXS) using JED-2300 instrument: 6490(LA). M–H loops were measured at room temperature using a vibrating sample magnetometer (VSM) Model Lake Shore, new 7400 series, USA.

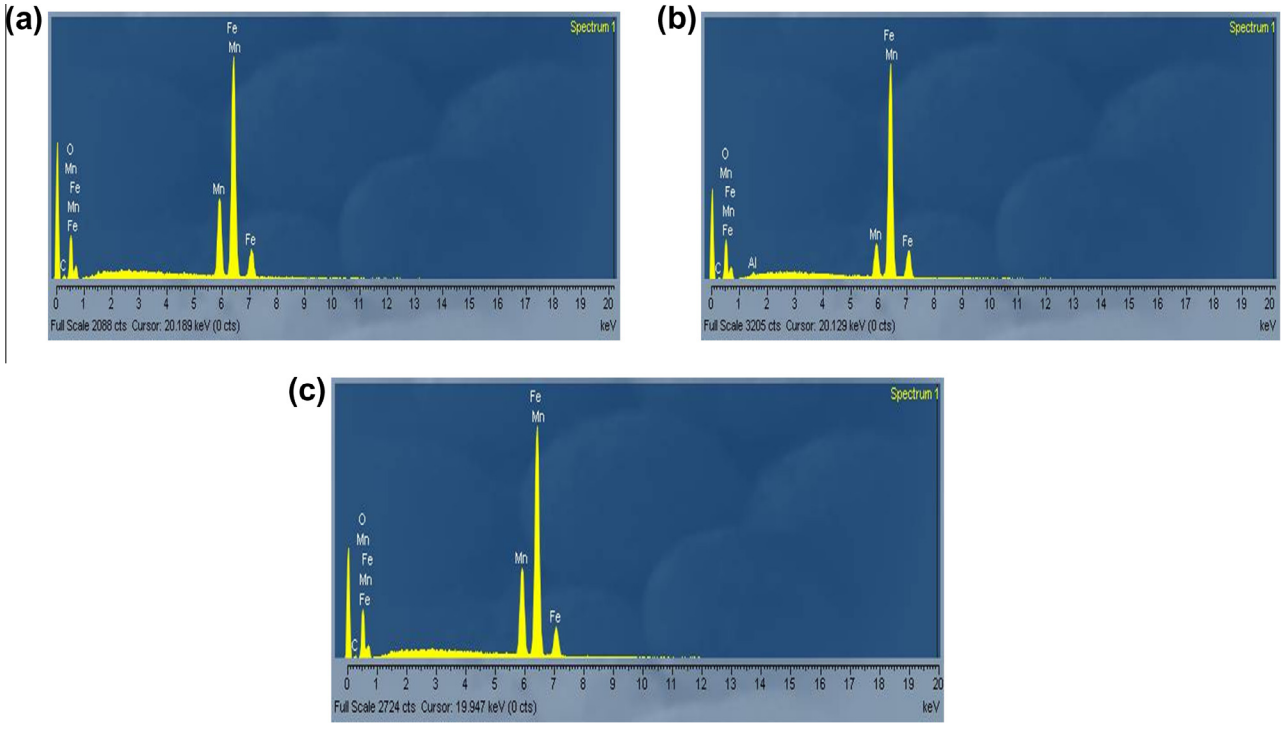

Fig. 2. EDX spectra (a–c) vs Mg-concentration for representative $Mn_{1-x}Mg_xFe_2O_4$ (x = 0.0, 0.3, 0.5) ferrites.