Available online at www.sciencedirect.com

ScienceDirect

Ceramics International 40 (2014) 227–232

CERAMICS INTERNATIONAL

www.elsevier.com/locate/ceramint

Synthesis and optimization of cubic $NiFe_2O_4$ nanoparticles with enhanced saturation magnetization

Mohammad W. Kadi[a], R.M. Mohamed[a,b,*]

[a]Department of Chemistry, Faculty of Science, King Abdulaziz University, P.O. Box 80203, Jeddah 21589, Kingdom of Saudi Arabia
[b]Advanced Materials Department, Central Metallurgical R&D Institute, CMRDI, P.O. Box 87, Helwan, Cairo 11421, Egypt

Received 12 May 2013; received in revised form 30 May 2013; accepted 31 May 2013
Available online 6 June 2013

Abstract

Single phase $NiFe_2O_4$ nanoparticles were prepared via the organic acid precursor method. XRD, FTIR, SEM, and magnetization techniques were utilized to determine optimal conditions to prepare $NiFe_2O_4$ with desired properties. Calcination for 2 h at 600 °C was optimal to produce the cubic particles. The best Fe/Ni molar ratio was determined to be 2:1.1. Oxalic acid was selected as the precursor acid. Under these conditions, cubic particles with average sizes of 46 nm were obtained. A saturation magnetization value of 61.8 amu/g and a coercivity of 221.5 Oe were observed for the particles at room temperature under optimized conditions.

Keywords: A. Precursors: organic; C. Magnetic properties; D. Ferrites

1. Introduction

Ferrites have numerous industrial applications. Their importance as semiconducting magnetic materials is clearly manifested in electric and magnetic devices such as transformers, memory chips, and spintronics [1–3]. Nickel ferrite ($NiFe_2O_4$) is a soft magnetic ferrite with an inverse spinel structure. In this structure, Ni^{2+} ions occupy octahedral sites, while Fe^{3+} ions are equally distributed between octahedral and tetrahedral sites [4]. Antiparallel spin-coupling between octahedral and tetrahedral sites causes the ferromagnetic behavior of this ferrite [5]. $NiFe_2O_4$ is extensively studied because of its favorable properties such as chemical stability, high magneto-crystalline anisotropy, high electrical resistivity, chemical stability, mechanical hardness and a promise of a variety of new useful applications [6].

Morphological, dielectric, and magnetic properties for $NiFe_2O_4$ are very dependent on the preparation method and experimental conditions [4,7,8]. Many studies have reported the preparation of $NiFe_2O_4$ nanoparticles using different routes, such as hydrothermal, sol–gel, mechanical, coprecipitation, and others. For example, 20 nm irregular particles were produced by electrochemical synthesis of $NiFe_2O_4$ [6]. Nanospheres with close to the saturation magnetization of the bulk $NiFe_2O_4$ were synthesized by a reverse emulsion-assisted hydrothermal preparation [9]. Nanorods with high coercivity were synthesized by polymer-assisted coprecipitation [10]. The dependence of magnetic properties on the temperature of samples prepared by chemical coprecipitation using stable Fe and Ni salts was studied by Maaz et al. [11]. $NiFe_2O_4$ nanoparticles have also been synthesized by various methods and then coated with certain compounds to enhance specific properties of the resulting composite [12,13].

In this work, we report the preparation of highly regular cube-shaped nickel ferrite particles with the organic acid precursor method. Magnetic properties of the prepared particles were optimized based on the following factors: calcination temperatures, duration of calcination, Ni/Fe molar ratio, and the use of various organic acids in the preparation.

2. Experimental

2.1. $NiFe_2O_4$ synthesis

All of the chemicals used in this study were used as received without any further purification. Fe $(NO_3)_3 \cdot 9H_2O$ (purity 99%)

*Corresponding author at: Department of Chemistry, Faculty of Science, King Abdulaziz University, P.O. Box 80203, Jeddah 21589, Kingdom of Saudi Arabia. Tel.: +966 26 952293; fax: +966 26 952292.
E-mail address: mhmdouf@gmail.com (R.M. Mohamed).

0272-8842/$ - see front matter © 2013 Elsevier Ltd and Techna Group S.r.l. All rights reserved.
http://dx.doi.org/10.1016/j.ceramint.2013.05.128

and Ni $(NO_3)_2 \cdot 6H_2O$ (purity 98.3%) were used as iron and nickel sources. The nickel ferrite powders were synthesized with the following steps: (i) an aqueous solution of (Fe^{3+}:Ni^{2+}) ions with molar ratios of 2:1 was added to a certain amount of oxalic acid based on the stoichiometric ratios of Fe^{3+}:Ni^{2+}; (ii) the solution was stirred and evaporated at 80 °C until a clear, viscous resin was obtained; (iii) the viscous solution was dried at 110 °C for 24 h; and (iv) the dried product was calcined at a rate of 10 °C/min in static air atmosphere at temperatures ranging from 400 to 1000 °C.

2.2. Characterization

All of the prepared samples were characterized throughout the course of the analysis to deduce the optimal conditions for obtaining the best required characteristics of the produced ferrite samples. The characterization techniques used are summarized in the following points:

A. Different phases of calcined catalysts were observed using powder XRD (Bruker axis D8) employing Cu Kα radiation (λ=1.540 Å). The average crystallite size of the powders was automatically estimated from the corresponding XRD data using the Scherrer equation:

$$d = B\lambda/\beta_{1/2} \cos\ \theta$$

where d is the average particle size of the material under investigation, B is the Scherrer constant (0.89), λ is the wavelength of the X-ray beam, $\beta_{1/2}$ is the full width at half maximum of the diffraction peak and θ is the diffraction angle [14].

B. Fourier transform infrared spectroscopy (FTIR) was conducted on a Thermo Electron Magna 760 using potassium bromide (KBr) (99% purity FTIR grade, Sigma-Aldrich), in which the prepared sample powders were added to KBr in a mass ratio of 1:100 and mixed into a homogeneous solution using a shaker for 20 s prior to compressing into pellets for measurement.

C. The magnetic properties of the synthesized ferrite powders were observed using a vibrating sample magnetometer (VSM; 9600-1 LDJ, USA) at room temperature in a maximum applied field of 15 kOe. Saturation magnetization (Ms) and coercivity (Hc) were determined form the observed hysteresis loops.

D. A scanning electron microscope (JEOL JSM-6400) with an applied potential of 15 kV was used to observe the morphology of calcined samples.

2.3. Optimization methodology

After synthesis, the $NiFe_2O_4$ samples were subjected to various experimental conditions to determine those that produced optimal morphological and magnetic properties. The first of these conditions was the effect of calcination temperature. In this step, oxalic acid with a molar ratio of 1 was used as a precursor acid. Using an Fe:Ni molar ratio of 2:1 and a calcination time of 2 h, samples were calcined at 400, 600, 800, and 1000 °C at increments of 10 °C/min in static air atmosphere temperatures. The best ferrite samples were produced at 600 and 800 °C and were therefore selected for further examination of the effect of the Fe:Ni molar ratio on the final product. The best molar ratio and calcination temperature were then used to analyze the effects of calcination time. Lastly, after optimizing each of the above parameters, different organic acids were examined. Full characterization of the produced ferrite was performed at each step of the optimization process.

3. Results and discussion

3.1. Calcination temperatures

To study the effect of calcination temperature on the properties of the prepared $NiFe_2O_4$, the material was subjected

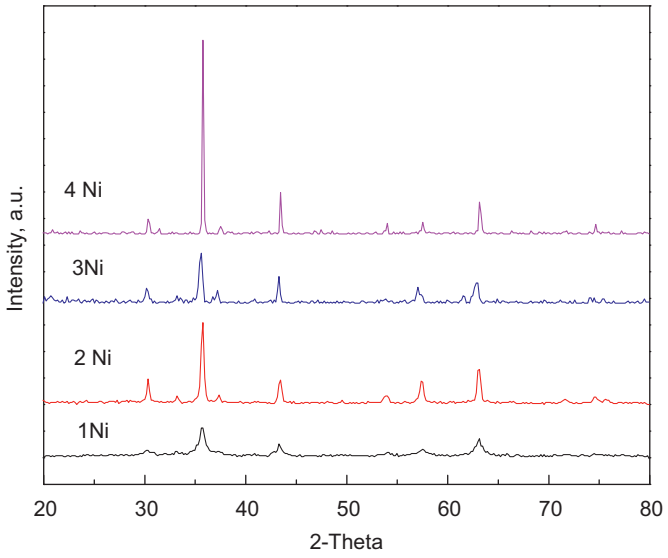

Fig. 1. XRD powder diffractograms for the calcined products.

Table 1
Applied calcination temperatures and observed properties of $NiFe_2O_4$.

Symbol of samples	Calcination temperature (°C)	Phases formed			Average particle size (nm)	Ms (emu/g)	Mr (emu/g)	Hc (Oe)
		NiO	Fe_2O_3	$NiFe_2O_4$				
1Ni	400	√	√	√	13.20	1.40	0.50	435.50
2Ni	600	√	–	√	46.40	39.20	18.50	856.80
3Ni	800	√	–	√	103.20	40.50	19.30	621.60
4Ni	1000	–	–	√	120.40	48.70	26.10	578.70