



# Size-dependent magnetic properties of calcium ferrite nanoparticles

Lavanya Khanna\*, N.K. Verma

Nano Research lab, School of Physics and Materials Science, Thapar University, Patiala-147004, India

## ARTICLE INFO

### Article history:

Received 31 May 2012

Received in revised form

8 January 2013

Available online 19 February 2013

### Keywords:

Nanotechnology

Nanocrystalline particles

Calcium ferrite

Nanocapsules

Magnetic behaviour

## ABSTRACT

The union of nanotechnology with the other fields of science heralds the influx of many newer and better technologies, with the capability to revolutionize the human life. In the present work, calcium ferrite nanoparticles were synthesized by conventional sol–gel method and were characterised by X-ray diffraction, Transmission electron microscope, Vibrating sample magnetometer and Fourier transform infrared spectroscope. The synthesized nanoparticles were calcined at different temperatures and their magnetic behaviour was studied. The synthesized nanoparticles calcined at 900 °C were formed in the shape of capsules and exhibited mixed characteristics of ferrimagnetic and paramagnetic grains with magnetic saturation of 0.85 emu/g whereas nanoparticles calcined at 500 °C were spherical in shape and exhibited superparamagnetic characteristics with saturation magnetization of 37.67 emu/g.

© 2013 Elsevier B.V. All rights reserved.

## 1. Introduction

Ferrites are iron based oxides with technologically fascinating magnetic properties, making them a prominent category in magnetic materials. The ferrite particles in nano-regime with significant change of physical properties provide more advantages over the bulk ferrites [1]. With regards to the rapidly mounting field of nanotechnology, ferrite nanoparticles have been the core of extensive research pertaining to their widespread applications, be it biomedical, technological or industrial. Nanocrystalline particles of magnetic materials show substantially enhanced magnetic properties. The magnetic behaviour of ferrites is strongly influenced by the particle size [2]. As the particle size decreases below the critical diameter, the coercivity decreases due to thermal effects, which become dominant enough to demagnetize a previously saturated assembly of particles [3]. Ferrite nanocrystallites of Mn, Co, Ni, Mg, Zn, K, Ca have been synthesized by reverse micelle [4–7,2], co-precipitation method [8,9], sol–gel auto combustion [1], thermal decomposition [10] and polymeric precursor method [11].

The synthesis and formation mechanism of Ca-ferrite compounds in bulk form have been carried out using different methods such as polymeric precursor method [11], under controlled oxygen potentials [12], thermolysis [13] and with different molar ratios of precursors under varying temperatures [14]. Moreover, it has been reported that the phase transition in Ca-ferrite is related to Fe<sup>+3</sup> high-spin/low-spin transition [15–18]. Ca-ferrite compounds so far have extensively been explored in the optical memory devices,

steel making industry (as deoxidizer, desulfuration, and dephosphorization) [14], pigment [11] and absorbent of hydrogen sulphide (H<sub>2</sub>S) [12]. All the above mentioned studies have been carried out on bulk Ca-ferrites. Till date and to the best of our knowledge no report is available on the size-dependent properties of Ca-ferrite at nanoscale.

It is well known that reduction in size leads to drastic changes in properties of materials. Therefore, the structure and size of ferrite nanoparticles are the important parameters to study their magnetic behaviour. In this communication, we report the synthesis of Ca-ferrite nanoparticles by sol–gel method. The morphology, structure and magnetic properties of Ca-ferrites at nanoscale have been studied in order to gain information regarding their formation mechanism in the nano regime. Further, the effects of different calcination temperatures on their morphology, structure and magnetic properties have also been reported.

## 2. Materials and methods

### 2.1. Materials

All the chemicals used for synthesis were of analytical grade. Calcium nitrate (Ca (NO<sub>3</sub>)<sub>2</sub> · 4H<sub>2</sub>O), Ferric nitrate (Fe (NO<sub>3</sub>)<sub>3</sub> · 9H<sub>2</sub>O), and citric acid (C<sub>6</sub>H<sub>8</sub>O<sub>7</sub> · H<sub>2</sub>O), were purchased from Loba Chemie, India and ethylene glycol was purchased from sdfine chemicals, India and were used without any further purifications.

### 2.2. Synthesis

Conventional sol–gel method was employed for the synthesis of calcium ferrite nanoparticles [11]. Metallic citrate polymerization using ethylene glycol is the basis of this process [11]. 1 M solution

\* Corresponding author. Tel.: +91 175 239 3513;

fax: +91 175 236 4498/239 3002.

E-mail addresses: lavanshya@yahoo.co.in,

lavanya.khanna@thapar.edu (L. Khanna), nkverma@thapar.edu (N.K. Verma).

of calcium nitrate and 2 M solution of ferric nitrate were mixed. Metal nitrates served dual role of being a soluble cation source and the oxidant [1]. To this mixture, 2 M citric acid solution was added along with 5–7 ml of ethylene glycol. Citric acid provided the fuel for the combustion; served as a chelating agent to form complexes with metal ions, preventing the precipitation of hydroxylated compounds [1] and non-polymeric filler for size modification and porosity control of nano powders [19]. Citric acid can be removed by either heat treatment or thermal decomposition, without affecting the properties of the material [19]. The solution was constantly magnetically stirred at 80–90 °C. The viscous gel began frothing after the removal of water molecules from the mixture. During evaporation, the solution became viscous and finally formed a very viscous brown gel. On further heating dried gel was formed, it continued to burn in a self propagating combustion manner until all the gel was completely converted to a brown-coloured powder, which also indicated the completion of auto-ignition process. The decomposition reaction would not stop until the whole citrate complex was consumed. Polymerization, promoted by heating, resulted in a homogeneous resin in which metal ions were uniformly distributed throughout the organic matrix [11]. The powder was thoroughly washed with ethanol and distilled water and was dried overnight in vacuum oven at 60 °C. The dried powder was further calcined at 300 °C (Ca3), 500 °C (Ca5), 700 °C (Ca7), 900 °C (Ca9), for 3 h each.

### 2.3. Characterizations

**Morphological and crystalline properties**—The morphology of synthesized Ca-ferrite nanoparticles (NPs) were studied by scanning electron microscope (SEM; JSM, 6510 LV, JEOL, U.S.A) where the dried sample was first coated with gold using JEOL, JFC sputter coater, energy-dispersive spectroscopy (EDS, Oxford) and transmission electron microscope (TEM; Hitachi (H-7500)), and the

crystalline properties were studied by X-ray diffraction (XRD; X'PERT PRO Panalytical, MRD ML).

**FT-IR analysis**—To study the bond formations, the FT-IR spectrum was recorded on Perkin Elmer Spectrum BX (II) spectrophotometer.

**Magnetic analysis**—The superparamagnetic properties and saturation magnetization of the synthesized Ca-ferrite NPs was determined using vibrating sample magnetometer (VSM; Princeton Applied Research Model 151/155).

## 3. Results and discussion

### 3.1. Crystalline and morphological properties

#### 3.1.1. XRD analysis

The room temperature XRD patterns were collected on X-ray diffractometer in the  $2\theta$  angle range of 10–70° with step size of 0.013° and average time of 13.77 s/step.

Fig. 1 shows the X-ray patterns of the uncalcined (as synthesized) and calcined nanoparticles at 300 °C (Ca3), 500 °C (Ca5), 700 °C (Ca7), 900 °C (Ca9). The uncalcined particles exhibited amorphous nature. No diffraction peaks were observed, depicting the lack of crystallinity in the uncalcined particles. Even on calcining at 300 °C, complete crystallinity was not obtained, as depicted by the X-ray pattern of Ca3 sample, only one peak at 35.3° was observed. The amorphous nature of uncalcined and Ca3 sample is also confirmed by the TEM micrograph in Fig. 3(a and b). The XRD patterns of calcium ferrite nanoparticles calcined at three different temperatures (500 °C, 700 °C and 900 °C) showed that all the calcined samples are crystalline in nature. On calcining the nanoparticles at 500 °C, spherical formation of nanoparticles was observed, this is also confirmed by the TEM micrograph in Fig. 3(c and d). In the XRD pattern, all the peaks for Ca5 are well

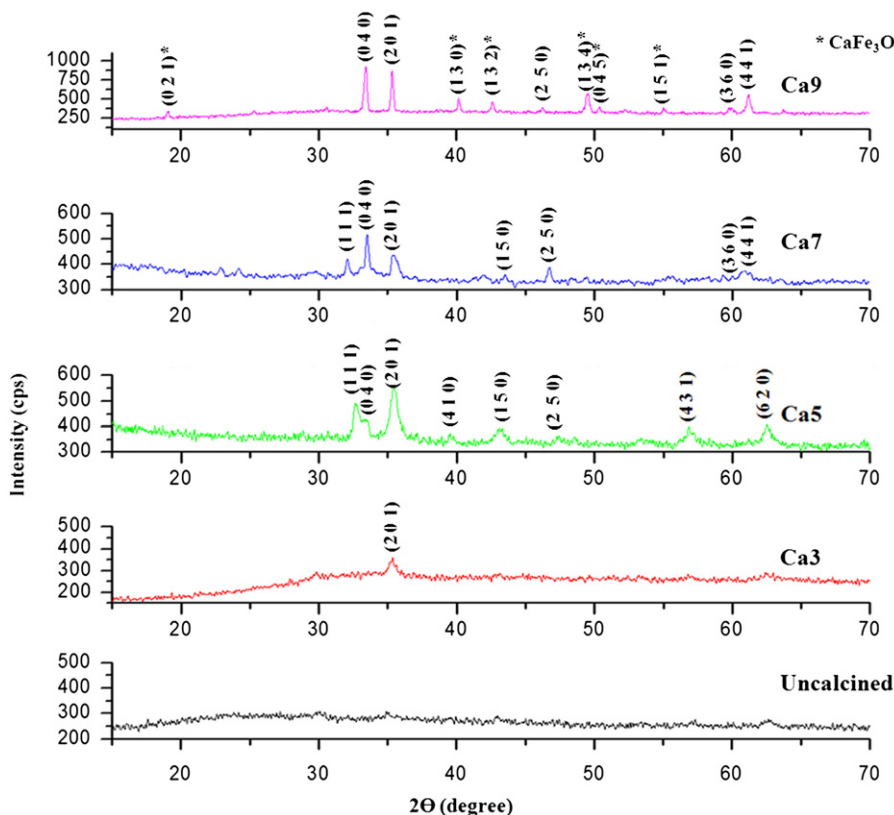


Fig. 1. XRD patterns of uncalcined and calcined Ca-ferrite nanoparticles.

Download English Version:

<https://daneshyari.com/en/article/8158682>

Download Persian Version:

<https://daneshyari.com/article/8158682>

[Daneshyari.com](https://daneshyari.com)