



Sonochemical assisted synthesis of SrFe₁₂O₁₉ nanoparticles



R.L. Palomino^a, A.M. Bolarín Miró^a, F.N. Tenorio^a, F. Sánchez De Jesús^{a,*}, C.A. Cortés Escobedo^b, S. Ammar^c

^aÁrea Académica de Ciencias de la Tierra y Materiales, UAEH, Carr. Pachuca-Tulancingo Km. 4.5, C.P. 42184 Pachuca, Hidalgo, Mexico

^bCentro de Investigación e Innovación Tecnológica del Instituto Politécnico Nacional, Cerrada Cecati s/n Col Sta. Catarina, C.P. 02250 Azcapotzalco, D.F., Mexico

^cITODYS, UMR-CNRS 7086, Université de Paris-Diderot, 75205 Cedex 13 Paris, France

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ABSTRACT

We present the synthesis of M-type strontium hexaferrite by sonochemistry and annealing. The effects of the sonication time and thermal energy on the crystal structure and magnetic properties of the obtained powders are presented. Strontium hexagonal ferrite (SrFe₁₂O₁₉) was successfully prepared by the ultrasonic cavitation (sonochemistry) of a complexed polyol solution of metallic acetates and diethylene glycol. The obtained materials were subsequently annealed at temperatures from 300 to 900 °C. X-ray diffraction analysis shows that the sonochemical process yields an amorphous phase containing Fe³⁺, Fe²⁺ and Sr²⁺ ions. This amorphous phase transforms into an intermediate phase of maghemite (γ-Fe₂O₃) at 300 °C. At 500 °C, the intermediate species is converted to hematite (α-Fe₂O₃) by a topotactic transition. The final product of strontium hexaferrite (SrFe₁₂O₁₉) is generated at 800 °C. The obtained strontium hexaferrite shows a magnetization of 62.3 emu/g, which is consistent with pure hexaferrite obtained by other methods, and a coercivity of 6.25 kOe, which is higher than expected for this hexaferrite. The powder morphology is composed of aggregates of rounded particles with an average particle size of 60 nm.

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1. Introduction

M-type hexagonal ferrites have been used extensively as permanent magnets and high-density magnetic recording media, among other interesting applications in recent decades; they are currently used in microwave devices at frequencies in the gigahertz range [1,2]. Although many other hard magnetic materials have been developed, the hexaferrite performance/cost ratio remains extremely favorable. Strontium hexaferrites are ferromagnetic materials with the formula MFe₁₂O₁₉, where M is the divalent Sr ion [3]. The magnetic properties of strontium hexaferrite depend on its structure and particle and crystallite sizes, which depend on the synthesis method used. This material possesses a high magneto-crystalline anisotropy (MCA), which occurs when the induced magnetization has a preferred orientation within the crystal structure. Because of their magnetization (~60 emu/g) and high coercivities (5.5 kOe) [3], strontium hexaferrites are commonly used as permanent magnetic materials in modern devices [4], such as loud speakers, permanent magnetic motors, coil instruments (galvanometer, voltmeter and ammeter) and microphones [7], as

well as being used to induce magnetic hyperthermia [5] and to generate hybrid photocatalysts [6].

SrFe₁₂O₁₉ has excellent characteristics because of its structure. As a typical magnetoplumbite type of hexagonal ferrite [8], Sr-hexaferrite is built from smaller units: a cubic block S with a spinel-type structure and a hexagonal block R that contains the Sr ions. The hexagonal Sr-ferrite has 24 magnetic Fe³⁺ ions per unit cell, which are distributed on five crystallographic sites [9]: three octahedral sites, 12 k, 2a and 4f₂; one tetrahedral site, 4f₁; and one trigonal bi-pyramidal site, 2b [10]. The chemical function has 12 Fe³⁺ ions, four of which have spins in the downward direction at 4f₁ (2 Fe³⁺) and 4f₂ (2 Fe³⁺), whereas the other 8 Fe³⁺ ions have spins in the upward direction at 12 k (6 Fe³⁺), 2a (1 Fe³⁺) and 2b (1 Fe³⁺). Because of its complicated crystal structure, the synthesis of Sr-ferrite represents a major challenge regardless of the selected method, particularly when a nanoparticle is the target of the study [11].

Because of the interest in these materials, nanostructured hexaferrites have been obtained using different methods. The conventional and oldest one is by calcination and sintering of a mixture of oxides or carbonates in a furnace at 1300 °C [12], but this technique produces large particles and consumes extensive energy. There are other synthesis methods that involve wet chemistry:

* Corresponding author.

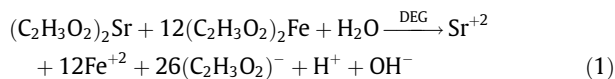
E-mail address: fsanchez@uaeh.edu.mx (F. Sánchez De Jesús).

co-precipitation [13], hydrothermal synthesis [14], mechano-synthesis [3,15] and sol-gel [16], among others. Recent studies have focused on different approaches to enhance the magnetic properties: substituting the cation positions in this type of structure [17–19], and synthesizing nanoparticles [20,21], which is ideal for magnetic performance. However, it is difficult to obtain ultra-fine and monodisperse particles by commercial ceramic methods because they involve high temperatures (>1300 °C) that produce unavoidable particle growth.

The use of ultrasound to produce nanoparticles has been a research topic of great interest in recent years [22–31]. In sonochemistry, molecules undergo a chemical reaction caused by the application of ultrasound radiation (20 kHz–10 MHz) [22]. This radiation induces the phenomenon of acoustic cavitation, which consists of the formation, growth, and implosive collapse of air bubbles in an irradiated liquid due to the relaxation and compression of a liquid solvent. These bubbles implode and cause shock-waves, which structurally modify the ultrasound-irradiated items [23]. Cavitation can produce a temperature of 5000 K over the course of a nanosecond, large cooling rates (1011 Ks⁻¹) and pressures greater than 1800 kPa [24]. The sonochemical route has emerged as a potential alternative to synthesize ferrites of nanometric size [25–28]. Recently, Junliang et al. [29] synthesized hexaferrite via ultrasonic irradiation, which was used to assist a co-precipitation method after calcination at 800 °C. Others studies have focused on demonstrating the influences of ultrasonic irradiation variables (power and time) on the shape and size of the synthesized ferrite nanoparticles [30–32]. Different studies have verified the ability of ultrasounds to induce chemical reactions, particularly through the formation of reactive radicals. In addition, the use of ultrasound is eco-friendly because all solvents can be reused, and it is more economic than other synthesis routes [22–28].

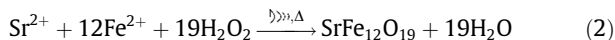
According to some authors [30–33], the synthesis of Sr-hexaferrite by ultrasonication a polyol solution can be represented as follows:

- a) The salts are completely dissolved in diethylene glycol (DEG) with a controlled quantity of water, and the hydrolysis of iron acetate in an aqueous medium ensues, according to Eq. (1):



Water is added to promote the hydrolysis of the metals, which is necessary to synthesize the oxides.

- b) After that, when the solution is sonicated, a strong oxidant (H₂O₂) is generated via the cavitation effect [24,30]. Then, the oxidant H₂O₂ is suggested to initiate the oxidation of Fe²⁺ to Fe³⁺ [32].
- c) Finally, annealing promotes the reaction between the metallic ions and H₂O₂ to yield strontium hexaferrite, according to the following reaction:



This work aims to produce SrFe₁₂O₁₉ nanoparticles using sonochemistry and a subsequent annealing at low temperature. This method is proposed as a simple, economical and eco-friendly synthesis for this type of nanomaterial. Moreover, the reaction mechanism behind Sr-hexaferrite nanoparticle formation was studied using the sonochemical-assisted method.

2. Experimental

To obtain SrFe₁₂O₁₉, stoichiometric amounts of (C₂H₃O₂)₂Sr (purity 99.995% Sigma Aldrich) and (C₂H₃O₂)₂Fe (purity 99.8%

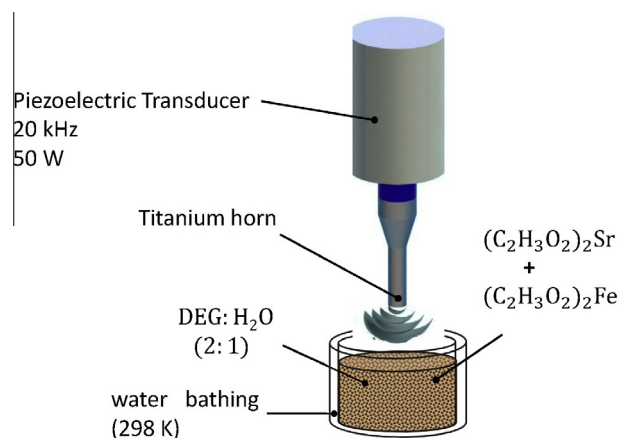


Fig. 1. Schematic representation of the sonochemical setup.

Sigma Aldrich), according to Eq. (2), were dissolved in 50 mL of a solution of diethylene glycol (DEG, C₄H₁₀O₃, purity 99.8% Sigma Aldrich) with water (weigh ratio of DEG:water was 2:1) for 30 min under mechanical stirring in air atmosphere. A solution without sonication was studied as a control experiment, as Sr²⁺ and Fe²⁺ ions were completely dissolved for 30 min of stirring time, solution was stirred for 5 h until observation of particles in suspension. The solutions were subjected to different sonication times from 10 min to 3 h using an Ultrasonic Homogenizer Q700 sonicator. Fig. 1 shows the schematic diagram of the sonochemical experiment setup. A water bath was used to control the reaction temperature. The solution temperature was kept at 25 °C during irradiation. After the sonochemical step, the powders were washed multiple times by forming a suspension in ethanol and centrifugation at 16,000 rpm (three times) for 15 min. Then, the powders were dried at 80 °C in air. At this point the powders were named “as obtained.” After that, the powders were annealed at different temperatures from 300 to 900 °C in air.

The obtained powders were characterized by X-ray diffraction (XRD) using an Inel Equinox 2000 diffractometer with Co Kα₁ (λ = 1.7890100 Å) radiation. The diffraction pattern was collected over a 2θ interval of 20–80° with increments of 0.02 (2θ). Rietveld refinements were performed on the X-ray diffraction patterns to obtain the percentages of different phases, crystallite sizes and microstrains of the powders. This method considers all of the collected information in a diffraction pattern and uses a least-squares approach to refine the theoretical line profile until it matches the measured profile [34]. Crystallographic data were obtained from the Crystallography Open Database (COD) [35] Scanning electron microscopy (SEM) using a JEOL-100-CX II was used to determine the morphologies and qualitatively characterize the particle sizes. The stabilities of the synthesized powders were measured by studying their thermal behaviors in a differential scanning calorimeter and a thermogravimetric analyzer (DSC/TGA 851e Mettler-Toledo). The experiments were performed with a heating rate of 10 Kmin⁻¹ using an air flow of 6 × 10⁻³ m³s⁻¹. Magnetization studies were performed at room temperature using a MicroSense EV7 vibrating sample magnetometer (VSM) with a maximum field of ±18 kOe.

3. Results and discussion

Fig. 2 shows the X-ray diffraction patterns of the powders obtained by applying 10 min of sonication and annealing at temperatures from 300 to 900 °C. Also, the XRD pattern corresponding to the powder obtained without applying sonication

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