

Synthesis and characterization of nanoparticles of CsFeO_2 using the sol-gel/combustion method

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ABSTRACT: Magnetic nanoparticles have many important applications in the technological, environmental, and medical fields due to their special properties (such as superparamagnetism). The main objective of this research is to obtain a magnetic ferrite. The sol-gel/combustion method was used to produce nanoparticles of cesium ferrite (CsFeO_2). Fourier transform infrared spectroscopy characterized the samples, indicating a ferrite phase at the peaks at 343 and 318 cm^{-1} and the metal-oxygen bond vibrations of the atoms in tetrahedral and octahedral sites.

KEYWORDS: Nanoparticles. Superparamagnetism. Cesium ferrite. Sol-gel/combustion. Fourier transform infrared spectroscopy.

RESUMO: As nanopartículas magnéticas possuem diversas aplicações de grande importância nas áreas tecnológica, ambiental e médica devido a propriedades especiais como o superparamagnetismo. O principal objetivo desta pesquisa consiste na obtenção de uma ferrita magnética. Foi utilizado o método de sol-gel/combustão para produzir nanopartículas de ferrita de césio (CsFeO_2). A espectroscopia no infravermelho por transformada de Fourier (FTIR) caracterizou as amostras, permitindo a observação da indicação da fase ferrita nos picos de 343 e 318 cm^{-1} , bem como das vibrações da ligação Metal-O dos átomos em sítios tetraédricos e octaédricos.

PALAVRAS-CHAVE: Nanopartículas. Superparamagnetismo. Ferrita de césio. Sol-gel/combustão. Espectroscopia infravermelha por transformada de Fourier.

1. Introduction

Current developments in nanoscience and nanotechnology have led to the discovery of numerous applications of magnetic nanoparticles in fields such as biomedicine, diagnostics, molecular biology, biochemistry, catalysis etc. Nanoparticles of magnetic oxides are mainly composed of Fe_2O_3 (hematite) [1].

Nanomaterials are a relevant topic of research due to their unique properties resulting from their small size. Ferrite nanoparticles notably stand out due to significant changes in their magnetic behavior as particle size decreases.

Nanometer-scale magnetic ceramic particles exhibit behaviors distinct from those of larger particles of the same material. By enhancing their properties, these particles can improve existing materials or even enable the development of entirely new materials. This has

spurred extensive research focused on understanding the properties of magnetic nanoparticles, especially those in the ferrite family, a group of metal oxides.

The first commercial ferrite products were developed in 1945 by Kato and Takeshi [2]. Since then, efforts have focused on manufacturing increasingly smaller and highly reliable components [3].

Cesium, the least electronegative element, has only one stable isotope, which was used in the synthesis of cesium ferrite (CsFeO_2).

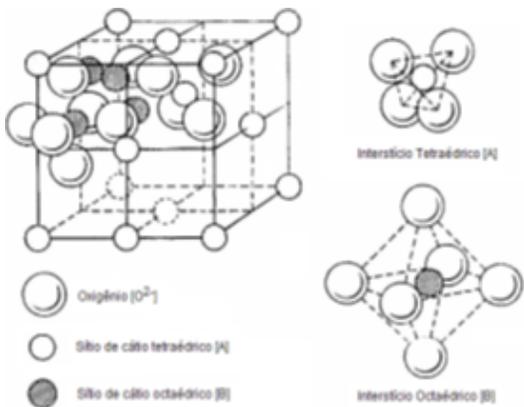
Several techniques, including combustion, sol-gel/combustion, and coprecipitation, can be used to prepare ferrite nanoparticles [4,5,6]. As such, the primary objective of this work is to obtain a magnetic ferrite. CsFeO_2 nanoparticles were synthesized using the sol-gel/combustion method and characterized via Fourier-transform infrared spectroscopy (FTIR), a fast instrumental technique that detects several functional groups in compounds.

1.1 Theoretical Basis

In order to understand the properties of ferrite, it is necessary to analyze the crystal structure of these materials. Metal oxides have the structure of the mineral spinel ($MgAl_2O_4$), in which the ions O^{2-} with atomic radius of 0.13 nm form a facecentered cubic lattice. Ions of smaller atomic radius occupy tetrahedral sites (Mg^{2+}) called sites [A] and octahedral sites [Al^{3+}], called sites [B] [7].

Ferrite has a spinel-like crystal structure, as shown in Figure 1, represented by the chemical formula $M^{2+}Fe_2^{3+}O_4^{2-}$, where M are divalent metals such as Mn^{2+} , Co^{2+} , Ni^{2+} , Cu^{2+} , Fe^{2+} , Zn^{2+} , and the occupation of tetrahedral and octahedral sites by divalent ions influences the magnetic properties of these materials [8].

Figure 1 - structure of the spinel.



Source: [6]

Since not all available sites are occupied, there are three different configurations that the structure can take: normal spinel, partially inverted spinel and inverted spinel. The normal spinel is defined as the configuration in which only the divalent metal ions (M^{2+}) occupy the sites [A] while Fe ions $^{3+}$ occupy the sites [B]. In the configuration with M^{2+} and Fe^{3+} at sites [A] and [B], with the ratio ranging from 0 to 1, the spinel is classified as partially inverted. In the configuration in which the Fe ions $^{3+}$ occupy the site [A] and half of the site [B], while the M ions $^{2+}$ occupy the other half of the site [B], the spinel is called inverted [7]. The occupation of the sites affects the magnetic behavior of the material.

This behavior is influenced not only by the crystalline structure but also by the macroscopic structure. One notable phenomenon observed in nanoparticles is superparamagnetism. Materials exhibiting this property do not retain magnetic memory, meaning that once they are removed from a magnetic field, no residual magnetization remains, provided the temperature is above the blocking temperature.

2. Development

The synthesis of $CsFeO_2$ powder using the sol-gel/combustion method [8] involved the following steps:

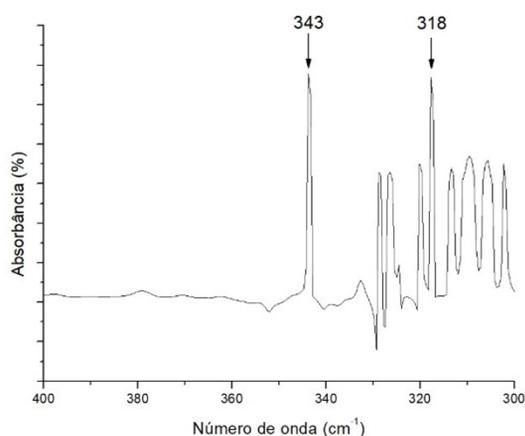
1. Preparing solutions of the chemical precursors $Cs(NO_3)_2 \cdot 6H_2O$ and $Fe(NO_3)_3 \cdot 9H_2O$ in distilled water at appropriate concentrations;
2. Preparing a solution of citric acid ($C_6H_8O_7 \cdot H_2O$) in distilled water at a molar concentration of 0.75 M;
3. Combining solutions 1 and 2 to obtain a homogeneous mixture;
4. Placing the solution obtained in step 3 on a hot plate at 60 °C and subjecting it to continuous stirring for approximately 4 hours to form a gel;
5. Heating the gel to 80 °C while maintaining stirring until it becomes transparent;
6. Increasing the temperature of the transparent gel to 200 °C to induce a self-combustion process, which occurs for about 20 minutes;
7. Dividing the material obtained in step 6 into several batches and subjecting them to heat treatments at 700 °C for 30 minutes, 1 hour, and 2 hours.

The infrared vibrational spectra were obtained using a Nicolet iS10 spectrophotometer via attenuated total reflectance (ATR-FTIR), with KBr as a dispersing agent, in the range of 400 to 300 cm^{-1} , at a resolution of 4 cm^{-1} , 32 scans, available at IPqM.

3. Results

In the infrared vibrational spectrum of the $CsFeO_2$ sample, as shown in Figure 2, ferrite phase indication is observed at peaks of 343 and 318 cm^{-1} , as well as the vibrations of the metal-oxygen bond, of atoms in the tetrahedral and octahedral sites [9,10].

Figure 2 - FTIR spectrum of cesium ferrite after a 1-hour heat treatment.



Source: [own elaboration].

4. Conclusion

This work shows that the sol-gel/combustion method is promising for obtaining cesium ferrite in the form of nanometric powder. The spectrum obtained by FTIR characterization demonstrates the success in synthesis with the observation of characteristic ferrite phase peaks and spinel-like structures.

Given the above result, the goal of this work was achieved, which was to obtain a magnetic ferrite.

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