

Cobalt Ferrite Nanostructures Obtained by Chemical Coprecipitation and Hydrothermal Method

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Abstract: Cobalt ferrite ($\text{Co}_x\text{Fe}_{3-x}\text{O}_4$) is a cubic ferrite with inverse spinel nanodimensional structure, its particles being very special ones due their magnetically interesting properties. Cobalt ferrite nanopowders were obtained using the chemical coprecipitation, an economical synthesis way of ultrafine $\text{Co}_x\text{Fe}_{3-x}\text{O}_4$ of different compositions (where $x = 0.25, 0.4, 0.5, 0.8$) and sizes (6-10nm) and hydrothermal method. We have obtained a precipitate with magnetic properties and color nuance varying from cinnamon to dark brown using the coprecipitation technique and brown-black by hydrothermal technique. The powders were characterized by XRD measurements. The main advantage of these two synthesis methods consist in dimensional control of the particle dimensions and form according to the desired magnetic properties. Besides hydrothermal way provides a higher degree of crystallinity and most pronounces magnetic properties.

Keywords: nanostructure, cobalt ferrite, coprecipitation, hydrothermal.

1. Introduction

Some properties of the ferrites can be enhanced by incorporation of divalent metallic ions inside their structure. Cobalt ferrites are spinels, and have the formula $\text{M}(\text{Fe}_2\text{O}_4)$, where M is the divalent cation cobalt (Co^{2+}). The oxygen anions (O^{2-}) adopt a close-packed cubic crystal structure, and the metal cations occupy the interstices in an unusual two-lattice arrangement. In each unit cell, containing 32 oxygen anions, 8 cations are coordinated by 4 oxygen (tetrahedral sites), and 16 cations are coordinated by 6 oxygen (octahedral sites).

The incorporation of cobalt ions results in an increase in saturation magnetization and in coercivity. Cobalt ferrites have high permeability and saturation magnetization, no preferred direction of magnetization, being "soft" magnetically (easily magnetized and demagnetized) and electrically insulating [1].

Cobalt ferrites are used in electrical equipment and electronic devices and are employed in high-frequency applications: telecommunications, quality filter circuits, high frequency transformers, wide band transformers, adjustable inductors, delay lines, and other high frequency electronic circuits, magnetic circuits for both low level and power applications. Some of the advantages of their utility are: low cost, shape versatility, high permeability, temperature and time stability, high electrical resistivity, resultant low eddy current losses over a wide frequency range (10 to 50 MHz).

The methods widely used for the synthesis of magnetic cobalt ferrite are: coprecipitation, hydrothermal, reverse

micelles, freeze-drying, spray roasting, and sol-gel processing [2]. In the present paper we describe the synthesis of cobalt ferrites by chemical coprecipitation and hydrothermal methods. The powders obtained are characterized by XRD diffraction, the hydrothermal synthesis pattern providing cobalt ferrite nanoparticles with better characteristics.

2. Method and samples

Chemical coprecipitation

The coprecipitation method was widely used to synthesize iron oxides. To obtain cobalt-ferrite nanoparticles, the next precursors were used: aqueous solutions of $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ and $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, mixed in stoichiometric proportion. Sodium Dodecile Sulfate was added then to obtain the micelle solution. The obtaining mixture was heated between 50 - 80°C by a magnetic stirring fitted with heating. The mixture pH was brought at values between 9.5 and 11 with NaOH 10% solution, the synthesis temperature being maintained at 70-95°C for 4-5 hours under vigorous magnetic agitation. The separation of particles was performed by decantation and filtration, then were washed with acetone on filter (paper) by 5-6 times and dried in air oven at 60°C for 2 hours.

Hydrothermal synthesis

Cobalt ferrite has been synthesized too using hydrothermal method that provides different classes of nanostructured anorganic materials from aqueous solutions, by means of small Teflon autoclaves. The synthesis conditions of $\text{Co}_x\text{Fe}_{3-x}\text{O}_4$ nanoparticles are: the

temperature up to 200°C and the pressure up to 100 bars. These pressure - temperature conditions facilitate the use of autoclaves with a simple structure that favours uniform particle obtaining. Were used as precursors: Iron nitrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$), Cobalt nitrate ($\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), Dodecile Sulfate ($\text{C}_{12}\text{H}_{25}\text{SO}_4\text{Na}$) – 0.3 moles, with molar ratio (Co/Fe) of 1:2, 1:3 and 1:4. The experimental procedure consists in separated by filtration and dried in oven at 80°C, obtaining a powder characterized by X ray diffraction.

3. Results and discussion

XRD spectra characteristic to those particles obtained by coprecipitation (see Fig.1) provides data considering to the particle size and degree of crystallinity. It shows not very pronounced peaks, fact that implies a lower degree of crystallinity of cobalt ferrite nanoparticles.

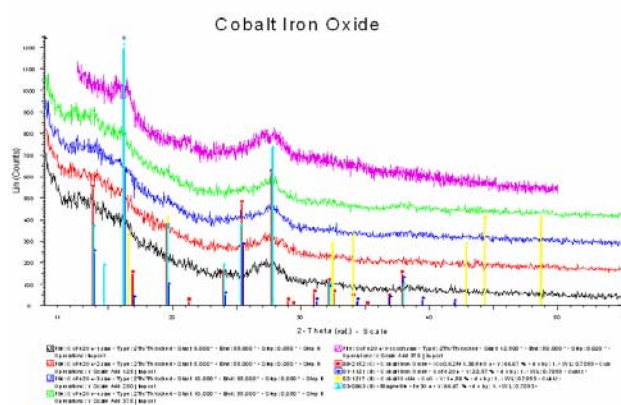


Figure 1. Diffraction X-ray pattern of cobalt ferrite nanoparticles obtained by coprecipitation method.

It is possible to obtain by hydrothermal method nanoparticles of $\text{Co}_x\text{Fe}_{3-x}\text{O}_4$ with high degree of crystallinity, desirable form and size, although the synthesis temperature is low, up to 200°C.

Particle's size represents the substance performance critical factor in monodisperse nanoparticles magnetic activity.

Figure 2 provides XRD spectra of cobalt ferrite powders obtained by hydrothermal method.

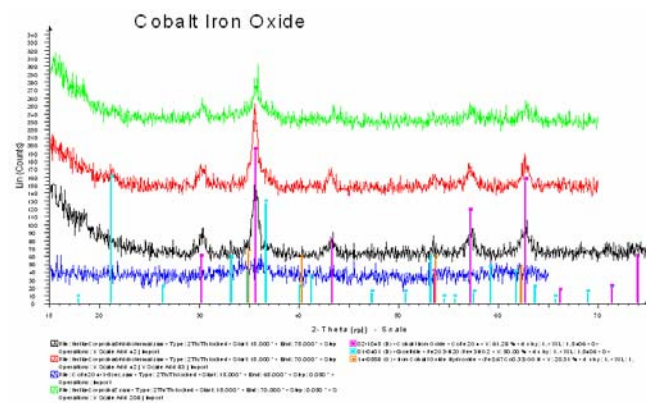


Figure 2. Diffraction X-ray pattern of cobalt ferrite nanoparticles prepared by hydrothermal method.

The most important attribute of hydrothermal synthesis consists in reduction of particle agglomeration attended to narrow size distribution. This method has a lot of benefits such as: clean product with high degree of crystallinity at a relative low reaction temperature (up to 200°C), form and dimension size controll, mono - dispersed particles.

We observe a brown-black powder and better results, namely a higher degree of crystallinity due higher diffraction peaks than coprecipitation specific ones and too much pronounced magnetic properties.

4. Conclusions

We had synthesized cobalt ferrite nanopowders by chemical coprecipitation and hydrothermal methods. Using the first method, the color of the obtained precipitate changes from cinnamon to dark brown according as temperature, pH values and reaction time. The material was proved to be magnetic. Also it was proved out that there is a tight connection between size and color of the particles, larger ones with more darkness color. It is easily to control the particle size according to magnetic properties.

Obtaining of a single phase asks for an adequate selection of pH value, reaction medium, synthesis time and crystallization temperature. The powder is not very well crystallized, small diffraction peaks in diffraction pattern.

Using the same precursors, the same pH and temperature values, by hydrothermal method we obtained cobalt ferrite nanoparticles with a higher degree of crystallinity as result from XRD patterns, much pronounced magnetic properties and a brown-black colour.

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