

A Solvent Free Synthesis and Characterization of Y-type Hexaferrite Particles in Presence of a Cationic Surfactant

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Abstract: Y-type Strontium magnesium hexaferrite with chemical composition $\text{Sr}_2\text{Mg}_2\text{Fe}_{12}\text{O}_{22}$ particles synthesized in presence of cationic surfactant- Cetyltrimethyl aluminium bromide (CTAB). Barium nitrate, magnesium nitrate, ferric nitrate and CTAB were used as raw materials. The viscous gel has been produced by mixing all materials at a constant temperature 80° – 100° C without any solvent and then calcined at 950° C for 6 hours in open air. The prepared hexaferrite samples were characterized by using various experimental techniques such as X-ray diffraction (XRD), SEM and Dielectric measurements. The XRD result shows the well crystalline phase of $\text{Sr}_2\text{Mg}_2\text{Fe}_{12}\text{O}_{22}$. The microstructure of prepared samples was observed via SEM. The dielectric measurements were carried out in order to study electrical characteristics of the materials, which proved useful in many research and development fields. Dielectric measurements of prepared hexaferrite samples were carried out using a LCR meter between 100 Hz to 2 MHz.

Keywords: $\text{Sr}_2\text{Mg}_2\text{Fe}_{12}\text{O}_{22}$ hexaferrite particles, XRD, SEM, solvent free process, structural properties

1. Introduction

Magnets have become an integral part of human endeavor, and are found in many industries such as automobile, telecommunication, data processing, electronics and instrumentation². The most used magnetic materials are ferromagnetic ceramics. Among the ceramics, hexagonal ferrites are mostly used because of their large number of technical applications compared to other materials. Ferrite is a member of a class of mixed oxides $\text{M}_x\text{Fe}_2\text{O}_3$, where M represents a metal such as cobalt, Manganese, nickel or zinc. The family of hexagonal ferrites can be classified on the basis of their chemical composition and crystal structure. They are subdivided into six types: M, W, Y, X, Z and U. The structure of these different types of hexagonal ferrites is

very complex and can be considered as a superposition of T, R and S blocks along the hexagonal c- axis, such as RSTSR*S*T*S* for Z-type, RSSR*S*S* for W-type, and RSR*S* for M-type, where T is a block of four layers of oxygen ions (O_4 -BaO₃-BaO₃-O₄) with composition Ba₂Fe₈O₁₄, R is a three oxygen layer block (O_4 -BaO₃-O₄) with composition BaFe₆O₁₁, S (spinel block) is a two oxygen layer block (O_4 -O₄) with composition Fe₆O₈, and the asterisk means that the corresponding block has been turned 180° around the hexagonal axis³. There is constantly increasing demand of signal processing devices because of mobile communications, security and defense. More over to these the operating frequencies are moving progressively higher from MW to millimeter waves, requiring use of hexaferrites. A solvent free route is an easy, rapid and environmental friendly route for the preparation of Y-type strontium magnesium hexaferrite. Synthesis of Sr₂Mg₂Fe₁₂O₂₂ hexaferrite required a simple mixing of chemical reagents at room temperature and then powder needs to dry at 80-100° C followed by final calcinations at 950° C. Conventional ceramic method needs long calcinations time and high temperature to produce a well crystalline Y-type Sr₂Mg₂Fe₁₂O₂₂ phase. In some way a solvent free route is modification of conventional ceramic method except the presence of cationic surfactant-CTAB, just which acts as an excellent flux and makes present route superior to the conventional ceramic method, including shortened reaction time, typically hexagonal structure with small particle size, etc.⁴. In present paper we report synthesis and Characterization of Y-type- Sr₂Mg₂Fe₁₂O₂₂ hexaferrite particles prepared by using a solvent free method in presence of a cationic surfactant- CTAB. The basic purpose of our investigations is to study the effect of CTAB on morphology and dielectric properties of Sr₂Mg₂Fe₁₂O₂₂ hexaferrite prepared by a solvent free synthesis route.

2. Experimental Section

(a) Sample preparation

Y-type strontium magnesium hexaferrite (Sr₂Mg₂Fe₁₂O₂₂) particles have been prepared from a mixture of strontium nitrate, magnesium nitrate, ferric nitrate and CTAB. Strontium nitrate [Sr(NO₃)₃ .6H₂O, > 99.0 % purity, Merck], magnesium nitrate [Mg(NO₃)₂.4H₂O], ferric nitrate [Fe(NO₃)₃.9H₂O, > 98 % purity, Merck] were used as starting materials. The mixture heated at constant temperature of 80° C for 2 hours and pre calcinated at 400° C followed by final calcinations at 950° C for 6 hours (fig.1). The same procedure was repeated using different amount of surfactant: cetyltrimethylammonium bromide (CTAB) 1 gm, 2 gm and 3 gm.

A different characterization methods⁴ used to test reliability of prepared samples. Samples were characterized in terms of morphology, grain size and crystalline structure using SEM and XRD measurements. Dielectric measurements on sintered samples were carried out at room temperature using LCR Bridge.

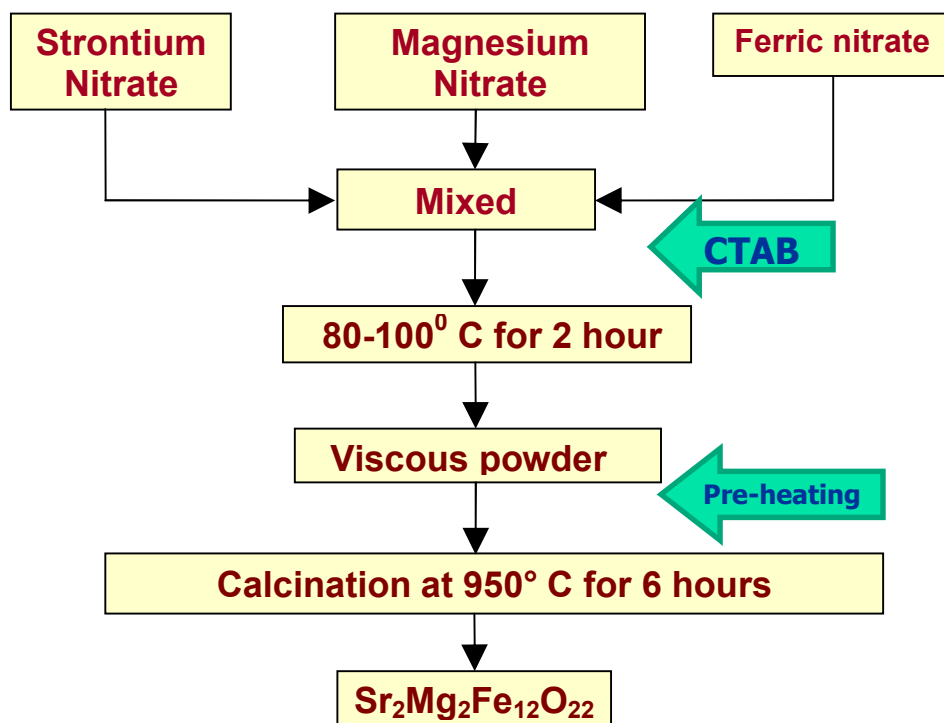


Figure1. Flow chart to prepare strontium magnesium hexaferrite ($\text{Sr}_2\text{Mg}_2\text{Fe}_{12}\text{O}_{22}$) particles by using a solvent free route

(b) Characterization

Prepared samples were characterized using a various experimental techniques like XRD; SEM. Powder X-ray diffraction (XRD) data were recorded on an X-ray diffractometer model no. SEIFERT XRD- 3000 PTS. Scanning electron microscope (SEM) images were obtained on the model LEO 440i. Dielectric measurement is carried out using a 'Hewlett Packard' 4284A LCR meter. Dielectric properties like dielectric constant and dielectric loss tangent of prepared hexaferrite samples were measured between frequency ranges of 100 Hz to 2 MHz using.

3. Results and Discussion

(a) X – ray diffraction analysis

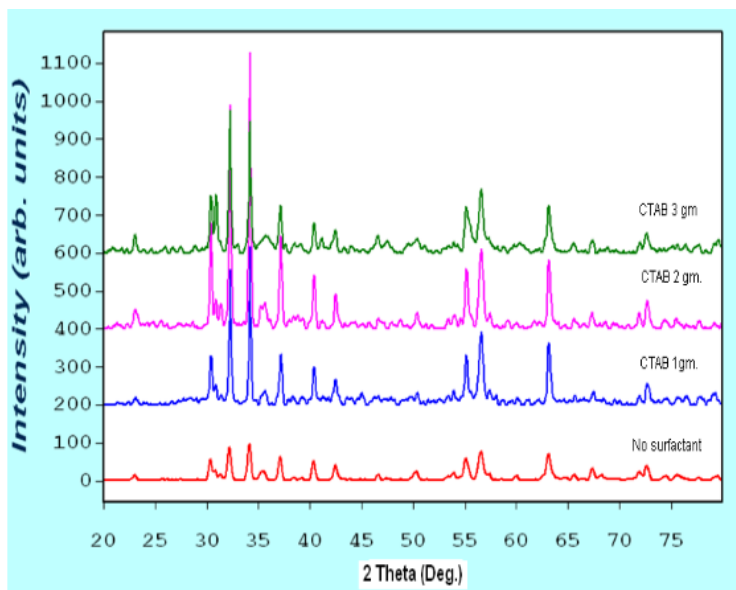


Fig. (2) XRD pattern of $\text{Sr}_2\text{Mg}_2\text{Fe}_{12}\text{O}_{22}$ particles prepared without and with different amount of CTAB (0 gm, 1 gm, 2 gm and 3gm.)

Fig. 2 represents XRD pattern of prepared hexaferrite samples. The X-ray diffractograms of samples calcinated at 950°C show Y-phase of $\text{Sr}_2\text{Mg}_2\text{Fe}_{12}\text{O}_{22}$ particles and Structure parameters a , c , cell volume V and c/a ratio for strontium magnesium hexaferrite samples (calcinated at 950°C for 6 hrs) are listed in Table 1. It is clear from Table 1 that with increase of the concentration of CTAB, lattice parameter ' a ' show slight increase in value and c/a ratio shows slightly lower value for normal sample (without CTAB) but remains constant for other (1 gm, 2 gm., 3 gm.) samples.

Sr. No	Samples	Lattice parameters		c/a	Cell volume $V (\text{\AA}^3)$
		a ($\text{\AA}$)	c ($\text{\AA}$)		
1	No surfactant	5.86	43.33	7.40	1303.98
2	CTAB 1 gm	5.99	43.68	7.39	1308.30
3	CTAB 2 gm	5.90	43.33	7.38	1300.31
4	CTAB 3 gm	6.21	43.77	7.03	1400.39

Table 1. Lattice constants (a , c), cell volume (V_{cell}) and c/a for strontium magnesium hexaferrite samples (calcinated at 950° C for 6 hrs)

(b) SEM study

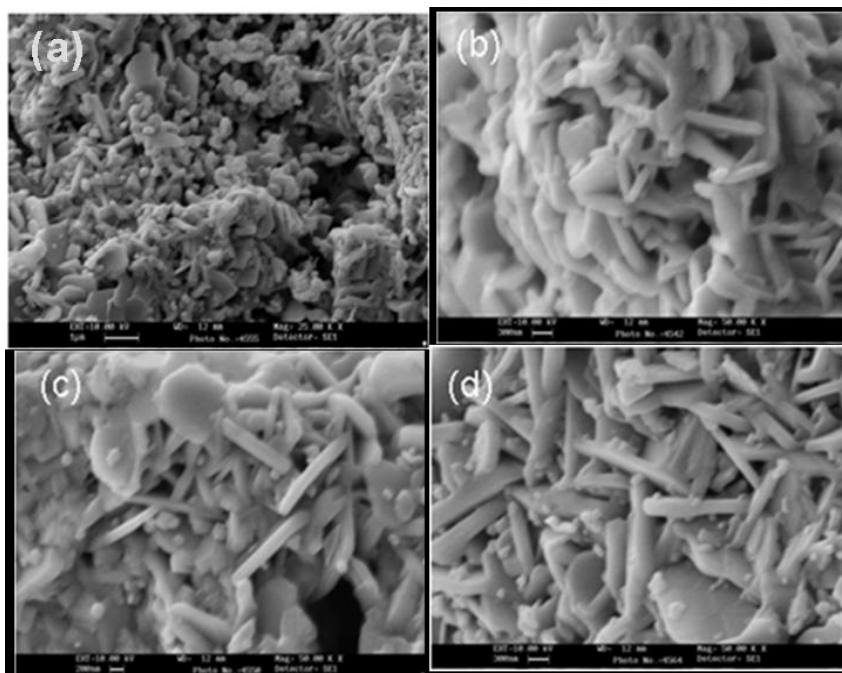


Fig. 3 SEM images of $\text{Sr}_2\text{Mg}_2\text{Fe}_{12}\text{O}_{22}$ particles with different amount of CTAB (a) 0 gm, (b) 1 gm, (c) 2 gm and (d) 3 gm

Fig 3 (a-d) shows the SEM images of $\text{Sr}_2\text{Mg}_2\text{Fe}_{12}\text{O}_{22}$ hexaferrites particles prepared with different amount of CTAB (0 gm, 1 gm, gm, 3 gm. respectively). In absence of CTAB fig. 3 (a) shows the formation of slight agglomerated hexagonal platelet structure of micro particle size but in presence of CTAB particles are found well crystalline and the hexagonal platelet structure is of nano particle size. It is observed that amount of CTAB plays a critical role in the morphology and particle size prepared Y-type $\text{Sr}_2\text{Mg}_2\text{Fe}_{12}\text{O}_{22}$ hexaferrite particles. SEM images show the formation of hexagonal platelet like structure.

(c) Dielectric measurements

In order to study dielectric properties of prepared hexaferrite samples, dielectric measurements were carried out at room temperature in frequency range of 100 Hz to 2 MHz. The results of frequency dependence of dielectric constant (real ϵ') and dielectric loss tangent for $\text{Sr}_2\text{Mg}_2\text{Fe}_{12}\text{O}_{22}$ hexaferrites with different amounts of surfactant are shown in Fig. 4 (a, b). It is clear

from fig. 4(a) as the frequency increases the dielectric constant (real- ϵ) gradually decreases and it attains a constant value at higher frequency. Fig. 4(b) shows the variation of the dielectric loss ($\tan \delta$) with frequency for different samples. The behaviour of $\tan \delta$ with frequency shows dielectric relaxation peaks, which shift towards higher frequencies with increasing amount of CTAB.

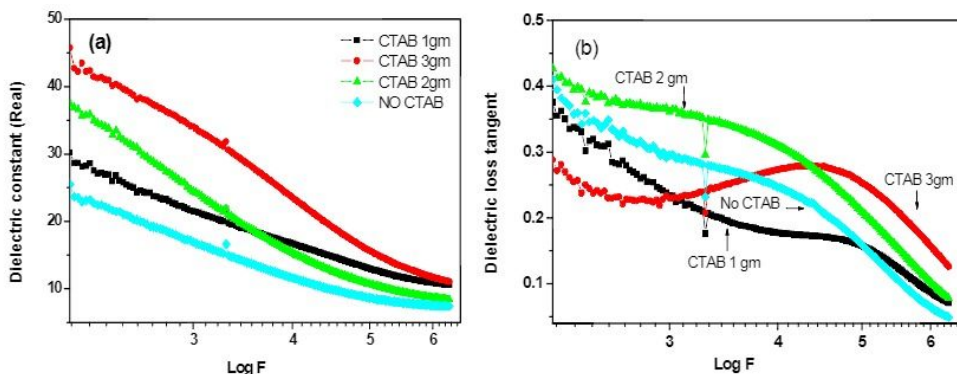


Fig. 4 (a, b) The variation of dielectric constant and dielectric loss tangent with frequency of $\text{Sr}_2\text{Mg}_2\text{Fe}_{12}\text{O}_{22}$ particles prepared without and with different amount of CTAB.

4. Conclusion

We have successfully synthesized Y-type $\text{Sr}_2\text{Mg}_2\text{Fe}_{12}\text{O}_{22}$ hexaferrite particles with and without presence of cationic surfactant. XRD result confirms the formation of well crystalline Y-phase. It is observed that amount of surfactant plays a critical role in deciding the morphology and particle size of prepared $\text{Sr}_2\text{Mg}_2\text{Fe}_{12}\text{O}_{22}$ hexaferrite particles.

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