

**MICROWAVE ASSISTED SOLUTION COMBUSTION SYNTHESIS AND
CHARACTERIZATION OF STRONTIUM HEXAFERRITE NANOPARTICLES**

Chandrashekhar A. Ladole*

Department of Chemistry, Shri Shivaji Arts, Commerce & Science College, Akot, 444101.

*Corresponding Author: Chandrashekhar A. Ladole

Department of Chemistry, Shri Shivaji Arts, Commerce & Science College, Akot, 444101.

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ABSTRACT

Ferrite nanoparticles have significant applications in the field of heterogeneous catalyst and actively participated in developments of nano-chemistry, chemical and material chemistry that deals with the development and additional level of understanding. In this study strontium hexaferrite nanoparticles ($\text{SrFe}_{12}\text{O}_{19}$) were synthesised by microwave assisted solution combustion synthesis. The resultant powder was investigated by powder XRD, FT-IR TG-DTA, SEM analysis. The existence of the single-phase perovskites structure with nano-crystalline size has been confirmed from the X-ray powder diffraction patterns. The stretching and bending vibrations of the metal cations are confirmed from the FT-IR spectra. The influence of ferrite synthesis condition, such as Sr/Fe molar ratio, pH value and time was investigated and powder was calcined at 500°C for 5 hours.

KEYWORDS: Strontium hexaferrite, nanoparticles, P-XRD, FT-IR, Microwave synthesis.**INTRODUCTION**

In recent years hard hexagonal ferrites such as $\text{SrFe}_{12}\text{O}_{19}$ have been a subject of continuous attention due to its applicability in magnetic memories, electronic components and recording media.^[1] The properties of a magnet of a hexagonal ferrite depend on the intrinsic magnetic properties of the M-type phase. Recently, magnetism in magnetic materials has been made much attention for many researchers due to its properties in magnetic information storage technology, cost effective magnetic materials and magnetic functional materials such as strontium ferrite.^[2-6] The discovery in the field of hexaferrite, tremendous efforts have been made in improving capabilities in magnetic materials and fundamental magnetic properties, crystallization of monodispersed flattened $\text{SrFe}_{12}\text{O}_{19}$ hexagonal shaped crystals and many studies have also been carried out concerning cationic substitution.^[7] Some hexaferrite used light rare earth ions as Pr, La and cation substitution for Sr and iron for ionic radius of elements.^[8] Considerable interest has been attracted in large number of synthetic techniques to control shape, properties and practical usefulness, particle size and high temperature ceramic properties. The synthesis of hexaferrite by different methods such as micro emulsion,^[9] auto-combustion^[10-11], sol-gel^[12-15], co-precipitation^[16], solid state reaction method^[17], ceramic process^[18-19] and ball milling.^[20] To obtain fine powder of hexaferrite, the precursor must be calcined at high temperature. Although many researchers have prepared strontium ferrite using co-precipitation technique which might lead to some disadvantages

including chemical in homogeneity, agglomeration. To prevent this disadvantage the sol gel auto combustion method is very useful, inexpensive and conventional to synthesize the strontium ferrite. Metal nanoparticles exhibit intensive interests in many fascinating physical properties and their applications in nano-devices, in organic chemistry reactions and considered to be very dynamic classes of nano magnetic materials with good electrical and dielectric properties and have been regarded as highly valuable electronic materials for more than 50 years.^[21-22] In the present work, we prepared $\text{SrFe}_{12}\text{O}_{19}$ by microwave assisted solution combustion method and studied properties of the $\text{SrFe}_{12}\text{O}_{19}$. The structural and morphological Characterizations were performed using, powder XRD, TG-DTA, FT-IR and SEM analysis.

Experimental work**MATERIALS AND METHODS**

All the chemicals with analytical grade were used and distilled before use whenever necessary. All the metal salts were obtained from SD fine chemicals and were used as received. Melting points were determined on a quality digital melting point apparatus. The FT-IR spectra were recorded on Bruker FT-IR spectrometer. The structural and phase purity of as prepared samples were characterized through XRD measurement using Bruker AXS-D8 advanced X-ray powder diffractometer with $\text{CuK}\alpha$ line ($\lambda = 1.54056 \text{ \AA}$) in the 2θ range from 10-90°. The morphology was studied using Scanning Electron Microscope (SEM) model (JEOL Model JSM -

6390LV) and the thermal analysis (TG/DTA) was carried out using a Perkin Elmer STA 6000 instrument.

Preparation of strontium hexaferrite

Strontium hexaferrite ($\text{SrFe}_{12}\text{O}_{19}$) was prepared by microwave assisted solution combustion method. The starting materials used were strontium nitrate and iron nitrate. The stoichiometric amount of $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ and $\text{Sr}(\text{NO}_3)_2$ was dissolved in double distilled water. The solution of citric acid was then added to the mixture of nitrates solution, to maintain the molar ratio of solutes in the solution as $\text{Sr}^{2+}:\text{Fe}^{3+}:\text{C}_3\text{H}_4(\text{OH})(\text{COOH})_3 = 1:12:19$. The precursor of citrate solution was stirred for half an hour to obtain the homogeneity. Finally, the pH 7of solution was adjusted using NH_4OH and calcined in

the muffle furnace at 500°C for 4 hours at a heating rate of $10^\circ\text{C}/\text{min}$ to obtained strontium hexaferrite powder.

RESULTS AND DISCUSSION

The XRD pattern recorded for strontium hexaferrite catalyst was obtained by solution combustion method and calcined at 500°C shown in Figure 1. The X-ray diffraction patterns of strontium hexaferrite supports well to the structural, and phase purity of the material. The indexed XRD peaks and sharp lines are observed due to crystalline a nature which is matched with standard data (JCPDS Card No: 84-1531), that further confirmed the hexagonal structure of the synthesized samples. The particle size was measured by Scherrer's formula and average particle size value is calculated as 28.82 nm.

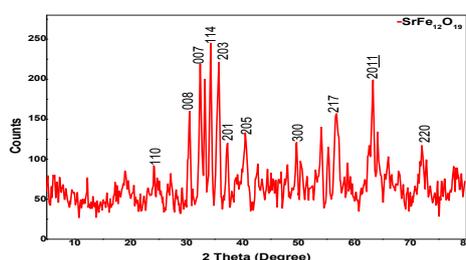


Figure 1: XRD spectra of $\text{SrFe}_{12}\text{O}_{19}$

Table 1: The parameters of units cell, and observed & calculated X-ray diffraction data of strontium ferrite.

System: hexagonal $\Lambda = 1.5406$.

$A = 41.12$ $C = 650.53a = 13.8716A$. $c/a = .2177$ $V = 503.28$

| Peak no | d(Obs.) | d(Cal.) | I(Obs.) | Ind. Wt. | (h k l) | $\text{Sin}^2\theta \cdot E4(\text{Obs.})$ | $\text{Sin}^2\theta \cdot E4(\text{Cal.})$ | $2\theta(\text{Obs.})$ | $2\theta(\text{Cal.})$ |
|---------|---------|---------|---------|----------|---------|--|--|------------------------|------------------------|
| 1 | 2.9290 | 2.9290 | 145 | 1.0 | 1 0 1 | 691.6 | 691.6 | 30.50 | 30.50 |
| 2 | 2.7560 | 2.7560 | 219 | 1.0 | 3 2 0 | 781.2 | 781.2 | 32.46 | 32.46 |
| 3 | 2.6920 | 2.6983 | 200 | 1.0 | 2 0 1 | 818.8 | 815.0 | 33.25 | 33.18 |
| 4 | 2.6110 | 2.6215 | 245 | 1.0 | 4 1 0 | 870.4 | 863.4 | 34.32 | 34.18 |
| 5 | 2.5100 | 2.5147 | 221 | 1.0 | 2 1 1 | 941.8 | 938.3 | 35.74 | 35.68 |
| 6 | 2.4120 | 2.4112 | 117 | 1.0 | 3 0 1 | 1019.9 | 1020.6 | 37.25 | 37.25 |
| 7 | 1.6600 | 1.6659 | 107 | 1.0 | 6 2 0 | 2153.3 | 2138.0 | 55.30 | 55.08 |
| 8 | 1.6210 | 1.6225 | 157 | 1.0 | 5 2 1 | 2258.2 | 2254.0 | 56.74 | 56.69 |
| 9 | 1.4680 | 1.4676 | 199 | 1.0 | 7 2 0 | 2753.4 | 2754.8 | 63.30 | 63.32 |

The FT-IR spectra of strontium hexaferrite in the range $450\text{--}4000\text{ cm}^{-1}$ are presented in Figure 2. For comparison of strontium hexaferrite obtained by solution combustion method attributed to metal oxygen modes due to shorter bond length of tetrahedral sites and longer bond length of in octahedral sites. From the FT-IR spectra show

adsorption bands at 994 and 1033 cm^{-1} due to presence of nitrate groups. The strong band at 1455 cm^{-1} can be assigned to the presence of carbonate groups and bands at around 598 , and 551 cm^{-1} corresponds to typical M-O adsorption bands assigned to $\text{SrFe}_{12}\text{O}_{19}$.

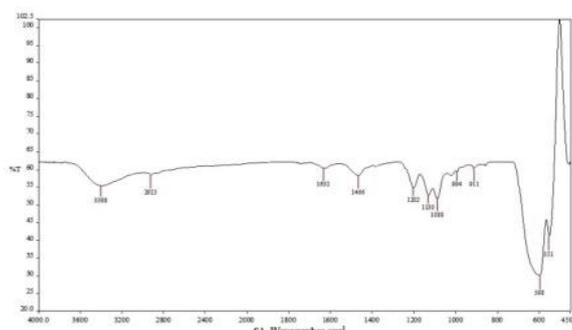


Figure 2: FT-IR of $\text{SrFe}_{12}\text{O}_{19}$

Thermogravimetric analysis TG/DTA curves of synthesized $\text{SrFe}_{12}\text{O}_{19}$ was shown in Figure 3 and determines the weight changes of a sample, whereas Differential Thermal Analysis (DTA) measures changes in temperature between a sample and a reference, as a function of temperature or time. (Perkin Elmer STA 6000). TG-DTA shows Fe, Sr and O elements identified. During heating chemical change of Strontium ferrite take

place. From the DTA curve endothermic peaks appear near 70°C and 90°C which caused due to desorption of water, while there is rapid increases in temperature from 100°C and again slightly decrease in temperature from 620°C up to 734°C . In TGA curve shows continuous weight loss up to 690°C . The total weight loss from room temperature to 720°C was about 86.53%.

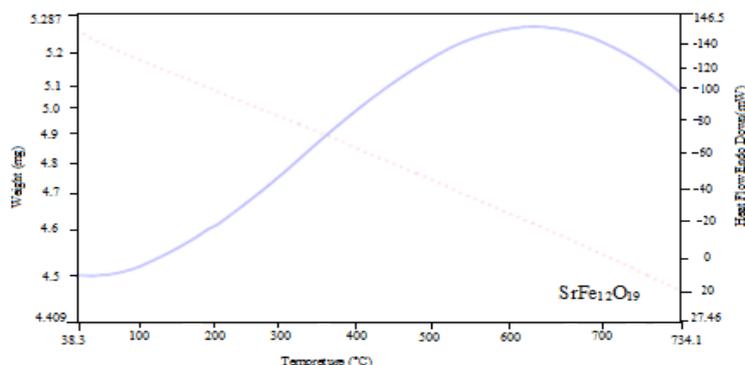


Figure 3: TG/DTA of $\text{SrFe}_{12}\text{O}_{19}$

The representative scanning electron micrographs of samples are shown in Figure 4. Surface analysis is used in understanding the surface features of the material. The shape of the material is very important for a variety of applications in different fields. In the present investigation an average grain size of about 1-10 μm found to be quite larger and this may be due to higher calcination temperatures for these M-type hexagonal

ferrites. Also larger grain size of material is due to higher microwave absorption. All samples appear chemically homogeneous, thus it is not possible to distinguish between $\text{SrFe}_{12}\text{O}_{19}$ and $\alpha\text{-Fe}_2\text{O}_3$ grains. The SEM images exhibiting agglomerated nanoparticles with non-regular shapes and this result agrees well with XRD data.

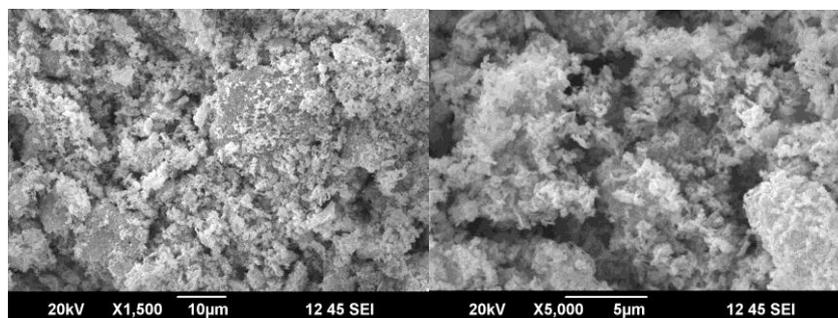


Figure 4: SEM images of $\text{SrFe}_{12}\text{O}_{19}$ hexaferrite.

CONCLUSIONS

Strontium hexaferrite material ($\text{SrFe}_{12}\text{O}_{19}$) was successfully prepared by microwave assisted an auto-combustion reaction process using citric acid and metal salts. The method was simple and cost effective. X-ray diffraction, Fourier transform infrared spectroscopy, and scanning electron microscopy analysis indicated that the prepared composites consisted of hard-magnetic phase ($\text{SrFe}_{12}\text{O}_{19}$), surface morphology shows agglomerated nano-particles with non-regular shapes. Thermogravimetric Analysis (TG) determines the weight changes of a sample, whereas Differential Thermal Analysis (DTA) measures changes in temperature between a sample and a reference, as a function of temperature or time. The influence of ferrite synthesis

condition, such as Sr/Fe molar ratio, pH value and time was investigated and powder was calcined at 500°C for 5 hours.

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