



# Factors controlling phase formation of novel Sr-based Y-type hexagonal ferrite nanoparticles

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**Abstract.** New Sr-based Y-type nanocrystalline hexagonal ferrites with a nominal chemical composition of  $\text{Sr}_2\text{Mg}_2\text{Fe}_{12}\text{O}_{22}$  ( $\text{Sr}_2\text{Y}$ ) were prepared by autocombustion from mixtures of  $\text{Sr}(\text{NO}_3)_2$ ,  $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  and  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ . The newly prepared  $\text{Sr}_2\text{Y}$  nanocrystalline particles were characterized by powder X-ray diffraction (XRD). A well crystalline phase of  $\text{Sr}_2\text{Y}$  with hexagonal crystal structure was observed. Fourier transform infrared spectroscopy (FTIR) studies revealed the information about the positions of the ions and their bonds within the lattice structure of the  $\text{Sr}_2\text{Y}$ . The chemical elements and their oxidation states in the  $\text{Sr}_2\text{Y}$  hexaferrites were determined using X-ray photoelectron spectroscopy (XPS). The XRD, FTIR and XPS studies confirmed the formation of  $\text{Sr}_2\text{Mg}_2\text{Fe}_{12}\text{O}_{22}$  hexaferrites. The morphology and porosity of the prepared  $\text{Sr}_2\text{Y}$  nanocrystalline  $\text{Sr}_2\text{Y}$  hexaferrite particles were studied by field emission scanning electron microscopy. The magnetic properties of  $\text{Sr}_2\text{Y}$  hexaferrites showed dependence on the methods of preparation conditions and calcination treatments. The values of coercivity, saturation magnetization and retentivity were in the range of 21.33–19.66 kA m<sup>-1</sup>, 42.44–38.72 emu g<sup>-1</sup> and 10.05–13.19 emu g<sup>-1</sup> respectively.

**Keywords.** Hexagonal ferrites; combustion process; X-ray photoelectron spectroscopy; glycine; oxidation state.

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## 1. Introduction

Ferrite-based magnetic materials have continued to attract attention due to their interesting magnetic properties. Recently, hexagonal ferrites have attracted more attention due to their use as permanent magnets [1]. Technological requirements of future applications including electronics devices, transformers and magnetic storage circuits require that material scientists continue to tune in the properties such as microstructure, homogeneity, particle shape and size, because such factors affect the extrinsic required magnetic behavior [2]. Among the various types of hexagonal ferrites, Y-type ferrites have proved to be the most promising candidates in magneto-optic recording devices, high-density recording media, spintronics, magnetic cell separation and microwave absorption devices [3,4].  $\text{Sr}_2\text{Y}$  comes under Y-type hexaferrite materials with the general formula  $\text{A}_2\text{Me}_2\text{Fe}_{12}\text{O}_{22}$  where A and Me refer to

divalent metal ions distributed among six sublattices (two tetrahedral and four octahedral).

Even though various techniques such as chemical coprecipitation, sol-gel synthesis, hydrothermal method, self-propagating high temperature and mechanical alloying have been adopted in the synthesis of hexagonal ferrites, the autocombustion synthesis is more cost effective where the entire reaction process takes 20 min. The autocombustion method provides a great opportunity for large-scale production of nanoparticles in a short span of time. This makes autocombustion a viable method of production for industrial manufacturing of ceramic materials.

Up to date, no studies were reported on  $\text{Sr}_2\text{Y}$  hexaferrite. In the present work, we shall report the synthesis of  $\text{Sr}_2\text{Y}$  hexaferrite by autocombustion using glycine as the fuel. The mechanism for the phase formation of  $\text{Sr}_2\text{Y}$  nanoparticles will be clearly presented and discussed. We have been able to produce an almost

pure single phase of  $\text{Sr}_2\text{Y}$  hexaferrites. The newly prepared nanocrystalline  $\text{Sr}_2\text{Y}$  hexaferrite particles were characterized by powder X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) to confirm the position of ions within the prepared  $\text{Sr}_2\text{Y}$  hexaferrites. The chemical elements and the oxidation states in the prepared nanocrystalline  $\text{Sr}_2\text{Y}$  hexaferrites were examined by X-ray photoelectron spectroscopy (XPS). The surface morphology was analysed by field emission scanning electron microscope (FESEM). The magnetic properties of the prepared  $\text{Sr}_2\text{Y}$  samples were measured by vibrating sample magnetometer (VSM). The preparation of the novel  $\text{Sr}_2\text{Y}$  hexaferrite nanocrystalline particles with reasonable magnetic properties will be demonstrated.

## 2. Experimental

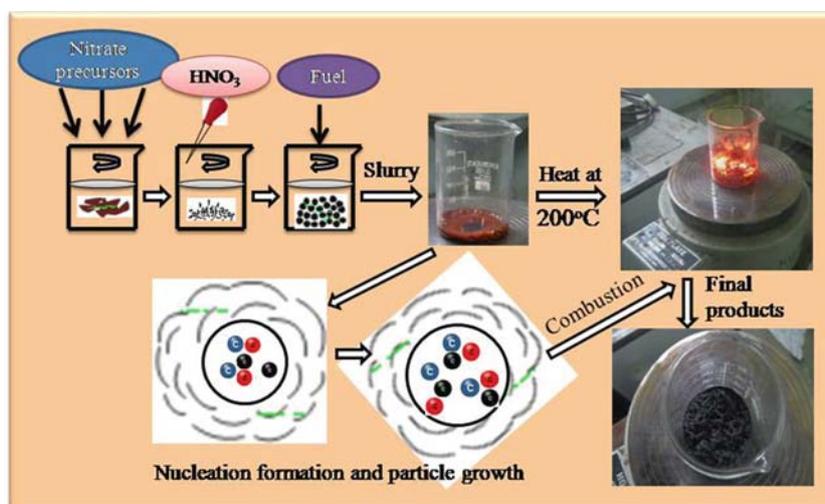
### 2.1 Synthesis

Hexagonal  $\text{Sr}_2\text{Y}$  ferrite nanocrystalline powders with a chemical composition of  $\text{Sr}_2\text{Mg}_2\text{Fe}_{12}\text{O}_{22}$  were prepared by the autocombustion method. The starting precursors were strontium nitrate ( $\text{Sr}(\text{NO}_3)_2$ , 99% purity), magnesium nitrate hexahydrate ( $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , 98% purity), iron (III) nitrate nonahydrate ( $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ , 98–100% purity) (all from Alfa Aesar), and glycine ( $\text{C}_2\text{H}_5\text{NO}_2$ , 98.5% purity) (from Qualigens). All analytical reagents were used without any further purification. Initially, stoichiometric amounts of strontium nitrate ( $\text{Sr}(\text{NO}_3)_2$ ), magnesium nitrate ( $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ) and ferric nitrate ( $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ) were dissolved in 20 ml of distilled water in a beaker, 10 ml of nitric acid ( $\text{HNO}_3$ ) was introduced into the beaker to get a

clear aqueous solution. The desired amount of glycine (fuel) was then added to the above solution, the whole mixture was stirred at a temperature of  $100^\circ\text{C}$  by a magnetic stirrer until the water slowly evaporated and the mixture became a dark brown viscous gel. This took about 2 h. The resulted dark brown viscous gel was kept on a hot plate at  $200^\circ\text{C}$  for about 10 min. By then the gel started to produce foams and sparks with the evolution of gaseous fumes. After the completion of combustion, a brownish powder was obtained. The time taken from actual ignition to the end of the reaction during the combustion process was less than 20 s. Finally, the as-prepared powder was grounded using an agate mortar and pestle to form a fine powder. A schematic diagram of the preparation step is given in figure 1.

### 2.2 Characterization

The crystal structure of the as-synthesized powders was identified by X-ray powder diffraction (XRD) measurements. XRD profiles were collected by Bruker D2 Phaser powder X-ray diffractometer with  $\text{CuK}\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ) over the range of  $10^\circ$  to  $80^\circ$  with a step mode of  $0.02 \text{ min}^{-1}$ . The as-synthesized powders were also analysed by FT-IR spectrophotometer (Bruker Alpha) using opus 6.5 (version) software. Small amounts of the as-synthesized powders were mixed with potassium bromide (KBr) and pressed into 1 mm thick transparent discs. IR spectra were collected from these discs. IR measurements were carried out at room temperature over the range  $350\text{--}1400 \text{ cm}^{-1}$ . Information about the oxidation states in



**Figure 1.** Schematic representation of  $\text{Sr}_2\text{Y}$  hexaferrite nanoparticles prepared by the combustion method.

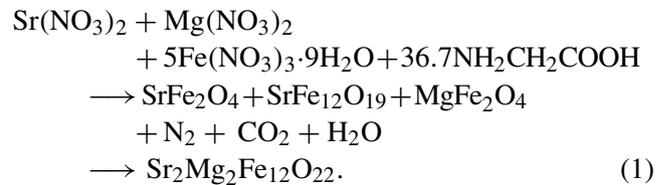
the as-synthesized powders was obtained from X-ray photoelectron spectroscopy (XPS). XPS studies were performed by Kratos Analytical Axis Ultra DLD with Al K $\alpha$ 1 source. X-ray photon of 1.486 keV with a pass energy of 160 eV was used for the survey spectrum with 40 eV narrow scans. The spectra were collected using a combination of electrostatic and magnetic lens (hybrid mode) for an analysed area of 700  $\mu\text{m} \times 300 \mu\text{m}$ . Surface charging effects were minimized using a charge balance operating at 3.6 V and 1.8 V maintained as filament bias. The angle between the normal to the sample surface and the direction of photoelectron collection are perpendicular to each other. Casa XPS software was used for peak fitting, and the procedures and conditions of the analyses were kept consistent for all the samples. In brief, a Shirley background was used, and the relative area, full-width at half-maximum (FWHM), Lorentzian–Gaussian (20% L–80% G) ratios, and peak positions were allowed to vary. The number of peaks for fitting was manually varied, and the minimum number of peaks that gave a reasonable fit were used for the analysis. The surface morphology was analysed by Carl Zeiss ULTRA 55 field emission scanning electron microscope (FESEM). The samples were coated with a thin gold layer before FESEM imaging. This enabled a resolution of  $\sim 1$  nm at an acceleration voltage of 5 kV. The working distance was 1 mm. The magnetic measurements have been carried out using Lakeshore 7410 vibrating sample magnetometer (VSM) with applied magnetic fields up to 10 kOe at room temperature.

### 3. Results and discussion

#### 3.1 Glycine-assisted combustion synthesis of $\text{Sr}_2\text{Mg}_2\text{Fe}_{12}\text{O}_{22}$

The nanocrystalline  $\text{Sr}_2\text{Y}$  particles were synthesized by autocombustion; however, their physicochemical properties depend on the nature and the amount of the fuel used. In order to prepare pure nanocrystalline  $\text{Sr}_2\text{Y}$  particles, glycine was utilized as the suitable fuel. It is cost-effective and can be easily obtained compared to other organic fuels. The autocombustion method depends on fuels that act as the reducing agents and metal nitrates that act as oxidizing agents. The reducing valencies of strontium, magnesium, iron, carbon and hydrogen are +2, +2, +3, +4 and +1 respectively and the oxidizing valencies of oxygen and nitrogen are –2 and 0 respectively. The complete redox reaction of

the metal nitrates with fuel (glycine) can be represented as

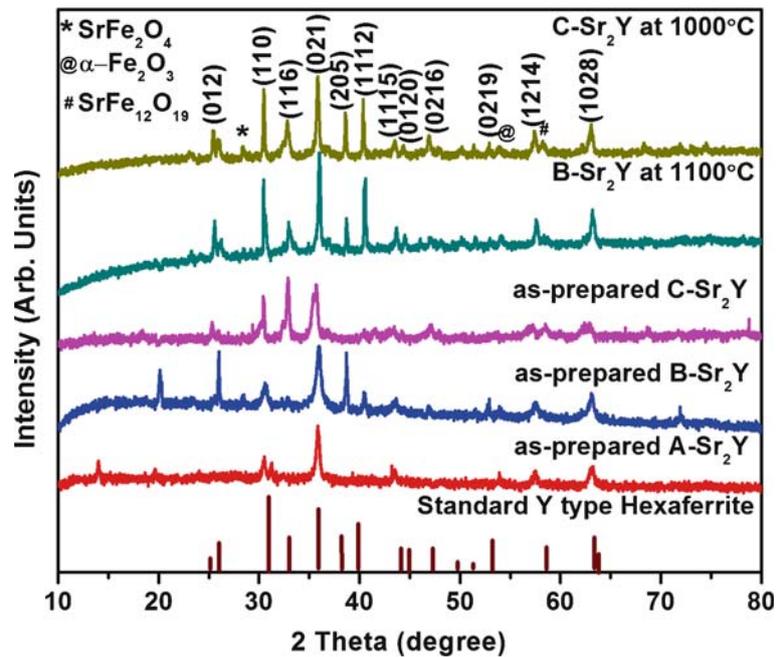


In this reaction, the metal nitrates mix with carbon and hydrogen from the glycine fuel to form gaseous products like  $\text{CO}_2$ ,  $\text{N}_2$  and water molecule. The total calculated valences of metal nitrates were –220 and +9 for glycine ( $\text{NH}_2\text{CH}_2\text{COOH}$ ). The stoichiometric composition of glycine–metal nitrates ( $\text{Gly}/\text{NO}_3^{3-}$ ) mixture becomes  $2 \times (-10) + 2 \times (-10) + 12 \times (-15) + n(+9) = 0$ , with  $n = 24.44$  mol for this redox reaction. During the formation of  $\text{Sr}_2\text{Y}$ , the number of moles of gases produced per mole of oxide is about 24.44. The calculated amount of glycine will be 36.7 g (assuming 0.5 M).

The autocombustion is an explosive method which yields many grams of powder within a short period of time even though a small amount of the powder will be lost in the high flames during the combustion. Therefore, safety precautions have to be taken. In this work, we have chosen fuel–lean composition with  $\text{Gly}/\text{NO}_3^{3-} < 24.44$  (glycine-to-nitrate ion molar ratio) which made the combustion reaction very mellow, and loss of powder in the high flames were greatly minimized. We estimated a loss of about 20% of the powder in the flames during the combustion process. For comparison, we used three different  $\text{Gly}/\text{NO}_3^{3-}$  molar ratios: 0.1818 (very fuel–lean reaction) and the powder produced is denoted as A- $\text{Sr}_2\text{Y}$ , 0.712 (fuel–lean reaction) and the powder produced is denoted as B- $\text{Sr}_2\text{Y}$  and 24.44 (fuel–stoichiometric reaction) and the powder produced is denoted as C- $\text{Sr}_2\text{Y}$ .

#### 3.2 X-ray diffraction studies

The X-ray diffraction (XRD) patterns of the prepared  $\text{Sr}_2\text{Y}$  nanocrystalline particles with different glycine-to-nitrate molar ratios are shown in figure 2. It can be seen that  $\text{Gly}/\text{NO}_3^{3-}$  ratio plays an important role in the formation and purity of the  $\text{Sr}_2\text{Y}$  nanocrystalline particles. For the A- $\text{Sr}_2\text{Y}$  sample, a mixture of phases had formed. The fuel might not be enough to mix completely with the metals and the combustion process was incomplete. By increasing  $\text{Gly}/\text{NO}_3^{3-}$  ratio (fuel–lean reaction), i.e., B- $\text{Sr}_2\text{Y}$ , a mixture of  $\text{Sr}_2\text{Y}$  hexaferrite and  $\text{Mg}_2\text{Fe}_2\text{O}_4$  was formed. Upon



**Figure 2.** X-ray diffraction pattern of  $\text{Sr}_2\text{Y}$  hexaferrite nanoparticles prepared by the combustion method.

the calcination of the B- $\text{Sr}_2\text{Y}$  powder at  $1100^\circ\text{C}$ ,  $\text{Sr}_2\text{Y}$  hexaferrite phase became more pronounced. However, the obtained  $\text{Sr}_2\text{Y}$  hexaferrite nanocrystalline particles were not completely pure and contained minute quantities of impurities. The XRD peaks were matched with the standard ICDD data card (44-0206) for the Y-type hexaferrites. The as-synthesized C- $\text{Sr}_2\text{Y}$  hexaferrite nanocrystalline particles (fuel-stoichiometric reaction) were much better than the B- $\text{Sr}_2\text{Y}$  nanoparticles in terms of the formation of the  $\text{Sr}_2\text{Y}$  hexaferrite phase. However, XRD peaks around  $36\text{--}40^\circ$  are absent and this suggests that the sample needs to be calcined at higher temperatures to bring out the Y-type hexagonal phase. Upon calcination at  $1000^\circ\text{C}$ , we obtained a nearly pure phase of  $\text{Sr}_2\text{Y}$  hexaferrite with minute quantities of impurities such as  $\text{SrFe}_2\text{O}_4$ ,  $\alpha\text{-Fe}_2\text{O}_3$  and  $\text{SrFe}_{12}\text{O}_{19}$ . Among the various stoichiometric compositions of glycine–metal nitrates mixture, the B- $\text{Sr}_2\text{Y}$  calcined at  $1100^\circ\text{C}$  and the C- $\text{Sr}_2\text{Y}$  calcined at  $1000^\circ\text{C}$  were found to be suitable for the formation of  $\text{Sr}_2\text{Y}$  hexaferrite nanocrystalline particles. The average crystallite size ( $t$ ) of the prepared  $\text{Sr}_2\text{Y}$  hexaferrites was determined by the X-ray line broadening method in accordance with the Scherrer equation [5]

$$t = 0.9\lambda / (\beta \cos \theta), \quad (2)$$

where  $\lambda$  (0.154 nm) is the wavelength of X-ray,  $\beta$  is the full-width at half-maximum (FWHM) and  $\theta$

is the Bragg's diffraction angle. This equation does not include any effects of the strains. The breadth of diffraction peaks in the XRD pattern were corrected for instrumentation broadening. The average crystallite size was found to be 45 nm for the  $1100^\circ\text{C}$  calcined B- $\text{Sr}_2\text{Y}$  hexaferrites and 53 nm for the  $1000^\circ\text{C}$  calcined C- $\text{Sr}_2\text{Y}$  hexaferrites. These values are comparable with the values of 56 nm (conventional sol–gel) and 34 nm (microwave heating) found for Y-type  $\text{Ba}_2\text{Co}_2\text{Fe}_{12}\text{O}_{22}$  [6].

The lattice constants for  $\text{Sr}_2\text{Y}$  hexaferrites were calculated from the following equation [7]:

$$1/d_{hkl} = [4/3(h^2 + hk + k^2/a) + l^2/c^2]^{1/2}, \quad (3)$$

where  $d$  is the value of  $d$ -spacing of the line in XRD pattern and  $(hkl)$  are the Miller indices. The calculated values of lattice parameters of the B- $\text{Sr}_2\text{Y}$  hexaferrites nanocrystalline particles calcined at  $1100^\circ\text{C}$  were  $a = 5.89 \text{ \AA}$  and  $c = 43.610 \text{ \AA}$  with an accuracy of  $\pm 0.002 \text{ \AA}$ , whereas for the C- $\text{Sr}_2\text{Y}$  hexaferrites calcined at  $1000^\circ\text{C}$ ,  $a = 5.97 \text{ \AA}$  and  $c = 43.703 \text{ \AA}$ . These values are in good agreement with that of  $\text{Ba}_2\text{Ni}_2\text{Fe}_{12}\text{O}_{22}$  ( $a = 5.859 \text{ \AA}$ ,  $c = 43.504 \text{ \AA}$ ) and  $\text{Ba}_2\text{Co}_2\text{Fe}_{12}\text{O}_{22}$  ( $a = 5.86 \text{ \AA}$ ,  $c = 43.504 \text{ \AA}$ ) in accordance with JCPDS Card No. 44-0206 [7].

The volumes of unit cells ( $V_{\text{cell}}$ ) of the calcined  $\text{Sr}_2\text{Y}$  hexaferrites were determined using the following equation [7]:

$$V_{\text{cell}} = a^2c \sin 120^\circ. \quad (4)$$

The calculated value of unit cell volume for the B- $\text{Sr}_2\text{Y}$  hexaferrites calcined at  $1100^\circ\text{C}$  was  $1325.134 \text{ \AA}^3$  and for the C- $\text{Sr}_2\text{Y}$  hexaferrites calcined at  $1000^\circ\text{C}$  was  $1310.546 \text{ \AA}^3 (\pm 0.0002)$ .

The X-ray density  $d_X$  of the calcined  $\text{Sr}_2\text{Y}$  hexaferrites were calculated using the following expression [8]:

$$d_X = ZM/N_A V_{\text{cell}}, \quad (5)$$

where  $Z$  is the number of molecules per unit cell,  $M$  is the molecular weight of the sample,  $N_A$  is the Avogadro's number  $= 6.023 \times 10^{23}$  and  $V_{\text{cell}}$  is the unit cell volume. The values of X-ray density were found to be  $6.273 \text{ g cm}^{-3}$  and  $6.201 \text{ g cm}^{-3}$  for the calcined B- $\text{Sr}_2\text{Y}$  and C- $\text{Sr}_2\text{Y}$  hexaferrite respectively. These values are comparable with the values of  $5.138 \text{ g cm}^{-3}$  for  $\text{Sr}_2\text{Co}_2\text{Fe}_{12}\text{O}_{22}$  and  $5.44 \text{ g cm}^{-3}$  for  $\text{Ba}_2\text{Ni}_2\text{Fe}_{12}\text{O}_{22}$  [7,9].

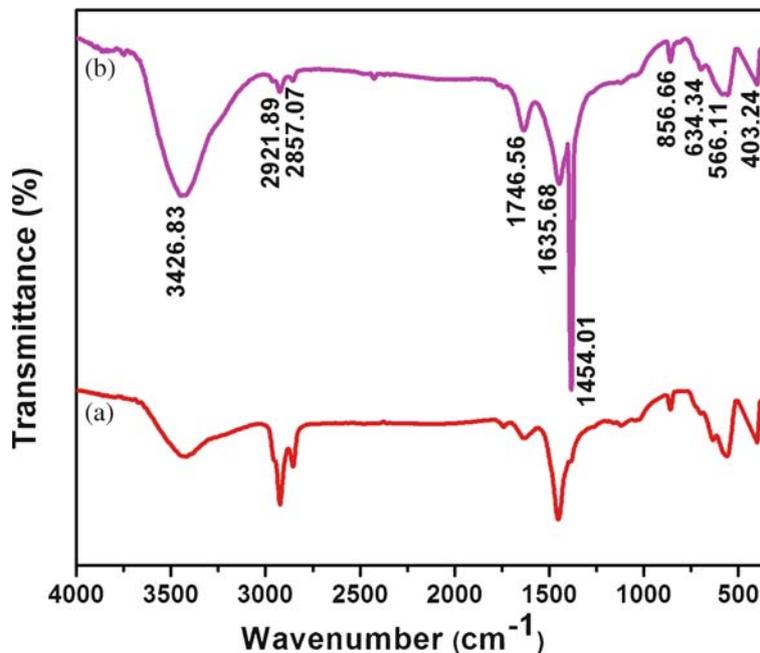
The specific surface area ( $S$ ) of the calcined  $\text{Sr}_2\text{Y}$  hexaferrite nanocrystalline particles was calculated from the X-ray density ( $d_X$ ) and the average crystallite size ( $t$ ) in accordance with the following equation [10]:

$$S = 6/d_X D. \quad (6)$$

The specific surface area was found to be  $18.046 \text{ m}^2 \text{ g}^{-1}$  for the calcined B- $\text{Sr}_2\text{Y}$  and  $21.459 \text{ m}^2 \text{ g}^{-1}$  for calcined C- $\text{Sr}_2\text{Y}$  hexaferrite nanoparticles.

### 3.3 FTIR analysis

Fourier transform infrared (FTIR) spectroscopy was used to gather information on the ions within the structure of calcined  $\text{Sr}_2\text{Y}$  hexaferrites. The FTIR spectra of the calcined B- $\text{Sr}_2\text{Y}$  and C- $\text{Sr}_2\text{Y}$  hexaferrites are shown in figure 3. The vibrational characteristics of one group of atoms within the network of different sublattices are usually independent of the vibrations of the other groups. This can provide significant information on the arrangement of atoms in hexaferrite particles. The characteristic IR absorption bands for the calcined B- $\text{Sr}_2\text{Y}$  are assigned as follows: the absorption band at  $3426.8 \text{ cm}^{-1}$  is attributed to the fundamental vibrational modes of the OH group and the two weak IR bands at  $2921.8 \text{ cm}^{-1}$  and  $2857 \text{ cm}^{-1}$  originate from the C-H stretching vibrations. The IR absorption bands at  $1746.5 \text{ cm}^{-1}$  and  $1635.6 \text{ cm}^{-1}$  can be attributed to the asymmetrical and symmetrical stretching vibrations of the  $\text{CO}_2^-$  group. The IR band at  $1454 \text{ cm}^{-1}$  can be attributed to the N-O stretching vibrations of  $\text{NO}_3^-$  (nitrate) group. The band at  $856.6 \text{ cm}^{-1}$  can be attributed to the vibrational frequency of the octahedral sites in the S and T blocks of the whole complex Y-type



**Figure 3.** FTIR spectrum of (a) B- $\text{Sr}_2\text{Y}$  hexaferrite nanoparticles at  $1100^\circ\text{C}$  and (b) C- $\text{Sr}_2\text{Y}$  hexaferrite nanoparticles at  $1000^\circ\text{C}$  prepared by the combustion method.

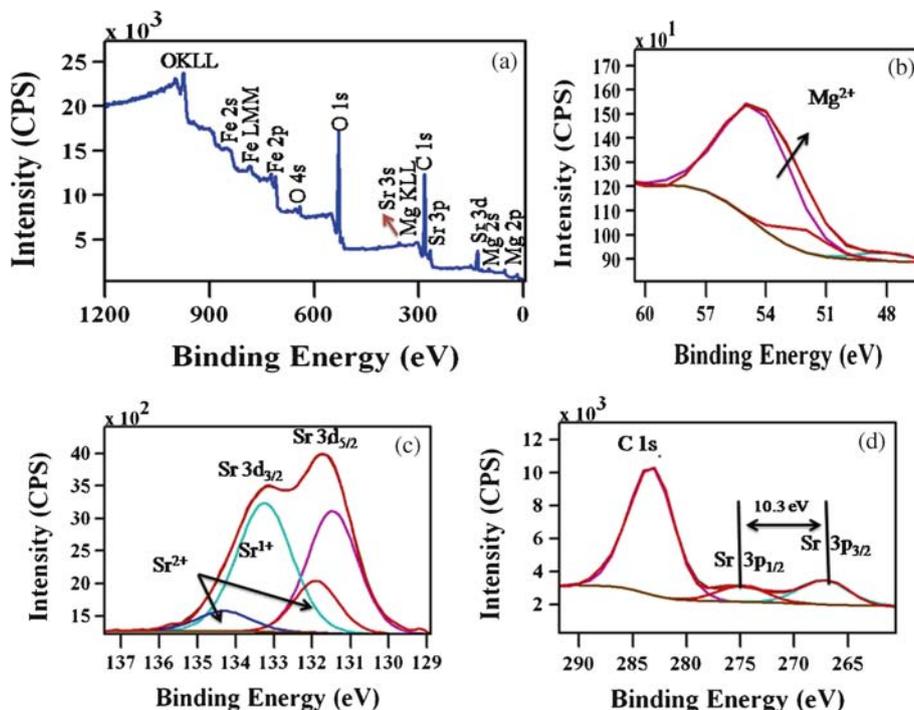
structure. The IR band at  $634.3\text{ cm}^{-1}$  is attributed to the octahedral sites in T-S block boundaries. The absorption band at about  $566.1\text{ cm}^{-1}$  is attributed to the octahedral group in spinel blocks. The band at  $403.24\text{ cm}^{-1}$  is attributed to the Fe–O stretching vibrations of the tetrahedral complexes in spinel blocks. Similar results were also observed for the calcined C-Sr<sub>2</sub>Y hexaferrite with small variations over the same range. These observations confirmed the chemical structure and positions of the ions within the prepared Sr<sub>2</sub>Y hexaferrite nanocrystalline particles. The IR results are consistent with crystal structure observed from the XRD measurements. The calcined Sr<sub>2</sub>Y hexaferrites have nearly single phase with minute impurity phases.

### 3.4 X-ray photoelectron spectroscopy studies

X-ray photoelectron spectroscopy (XPS) studies were performed to determine the chemical species and states of the elements in our samples. Figure 4a shows the wide spectrum of calcined C-Sr<sub>2</sub>Y hexaferrites nanocrystalline particles. The elements such as Sr, Mg, Fe, C and O were detected. No other elements were identified; this supports the nearly single phase obtained from the XRD analyses.

Figure 4c shows the Sr 3d XPS deconvolution spectra for the calcined nanocrystalline C-Sr<sub>2</sub>Y hexaferrites. The Sr 3d peak is split into two peaks, namely Sr 3d<sub>5/2</sub> and Sr 3d<sub>3/2</sub>, due to the spin orbital coupling. The Sr 3d<sub>5/2</sub> can be resolved into two consecutive peaks with binding energies of 131.9 eV and 132.6 eV which are assigned to Sr<sup>1+</sup> and Sr<sup>2+</sup> ions. The Sr 3d<sub>3/2</sub> can be resolved into two peaks at 133.6 eV and 134.8 eV corresponding to the binding energies of Sr<sup>1+</sup> and Sr<sup>2+</sup> ions. The difference of about 1.2 eV between the two peaks of Sr 3d<sub>3/2</sub> is attributed to Sr<sup>2+</sup> ion as reported in previous studies [11]. Figure 4d shows the Sr 3p XPS spectra for the C-Sr<sub>2</sub>Y hexaferrites calcined at 1000°C. The Sr 3p contains a C 1s peak. This may have resulted from minute quantities of impurities left in the sample from the combustion process. Based on spin-orbital splitting, the Sr 3p splits into two doublet peaks such as Sr 3p<sub>3/2</sub> and Sr 3p<sub>1/2</sub>. This doublet peaks are well deconvoluted with binding energy splitting of 10.3 eV corresponding to the Sr<sup>2+</sup> ion in its oxide environment [12,13].

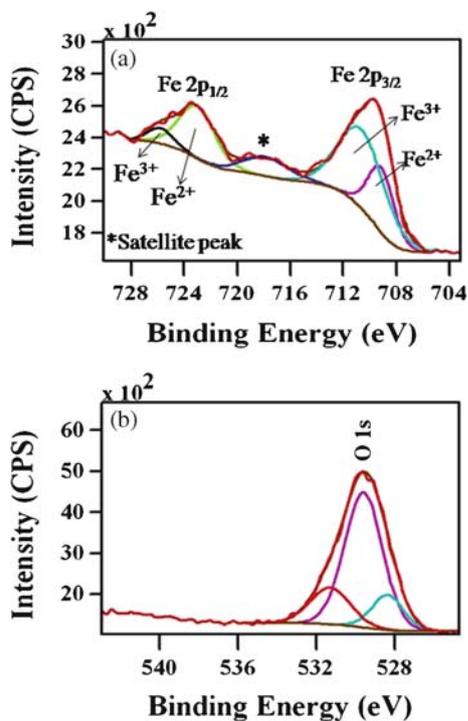
Figure 4b shows the deconvolution of Mg 2p spectra for the calcined nanocrystalline C-Sr<sub>2</sub>Y hexaferrites. These spectra are fitted with the raw data and deconvoluted to resolve the peaks. The Mg 2p core level is close to the valence band and the difference between



**Figure 4.** (a) XPS survey spectrum, (b) deconvolution spectra of Mg 2p peak, (c, d) high-resolution spectra of Sr 3d and Sr 3p peaks for C-Sr<sub>2</sub>Y hexaferrite nanoparticles at 1000°C.

Mg  $2p_{3/2}$  and Mg  $2p_{1/2}$  cannot be resolved. This is expected to reveal the charge and valence state of Mg more representatively. Gaussian fit was applied to the Mg  $2p$  peak and it was resolved into three distinct components. The minor peak at the lower binding energy of 49.5 eV is attributed to the metallic Mg which is in good agreement with previous reports [14,15]. The peak at 51.6 eV is assigned to MgB<sub>2</sub> [15]. The major peak at the higher binding energy of 54.4 eV is attributed to the divalent (Mg<sup>2+</sup>) species of Mg [15].

The Fe  $2p$  photoelectron peak of the calcined nanocrystalline C-Sr<sub>2</sub>Y hexaferrite particles is shown in figure 5a. Based on spin-orbit coupling, the Fe  $2p$  peak is split into Fe  $2p_{3/2}$  and Fe  $2p_{1/2}$  components. The Fe  $2p_{3/2}$  peak can be deconvoluted into two peaks at 709.14 eV and 710.60 eV corresponding to the binding energies of Fe<sup>2+</sup> and Fe<sup>3+</sup> ions. Similarly, Fe  $2p_{1/2}$  peak can be deconvoluted into two fine peaks at 723.02 eV and 725.78 eV attributed to Fe<sup>2+</sup> and Fe<sup>3+</sup> ions. However, a peak at 717.80 eV is present between the Fe  $2p_{3/2}$  and Fe  $2p_{1/2}$  components and it is considered as satellite peak. Due to large background, the chemical states of Fe are difficult to analyse. The difference of about 8.66 eV between the binding energies of the satellite peak and the principal peak of Fe  $2p_{3/2}$  is due to the presence of Fe<sup>3+</sup> ion



**Figure 5.** (a) High-resolution spectra of Fe  $2p$  peak and (b) XPS spectra for O  $1s$  peak for C-Sr<sub>2</sub>Y hexaferrite nanoparticles at 1000°C.

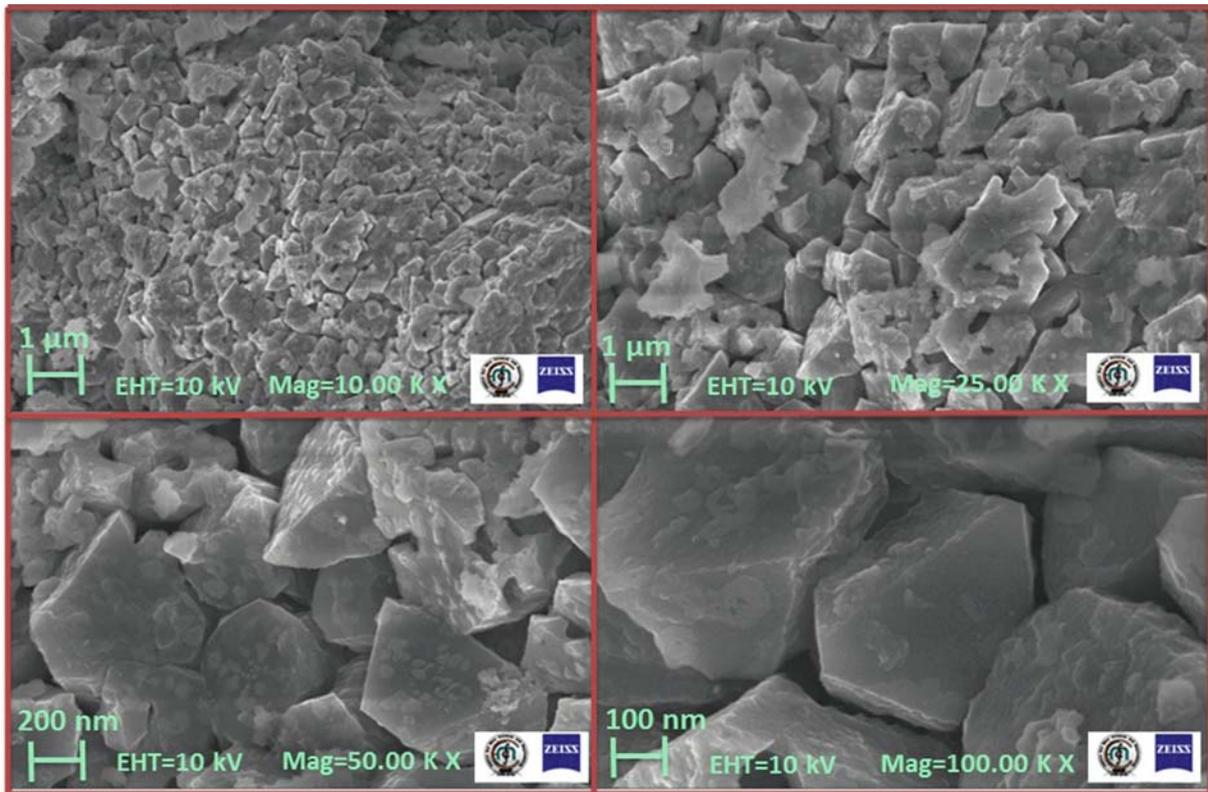
[16–18]. In principle, the valence of Fe ions fluctuate due to the reduction of Fe<sup>3+</sup> to Fe<sup>2+</sup> ions. In order to determine the presence of oxide/hydroxide species in the prepared samples, O  $1s$  spectra were analysed. Figure 5b shows the deconvolution of the spectra for the calcined C-Sr<sub>2</sub>Y hexaferrites. The O  $1s$  peak can be decomposed into three peaks. The first peak at 528.51 eV is attributed to lattice O<sup>2-</sup>, the second peak at 529.72 eV is assigned to the lattice OH<sup>-</sup> contribution from Fe(OH)<sub>2</sub> and FeOOH and the third peak at 531.33 eV corresponds to the adsorbed –H–O–H on the surface of the Sr<sub>2</sub>Y hexaferrites [19].

### 3.5 Morphology studies

The morphology of the prepared nanocrystalline hexaferrite particles were examined by FESEM. The FESEM images of the calcined C-Sr<sub>2</sub>Y hexaferrite nanoparticles at different magnifications are shown in figure 6. The Sr<sub>2</sub>Y hexaferrites have dense microstructure with uniform particle size distribution. Almost all the particles have hexagonal symmetry with definite crystalline grain boundaries. The shape of the grains in the hexaferrites determines the right technological application. Rod-shaped hexaferrites can be utilized in surface-enhanced Raman scattering, imaging, catalysis, data storage and sensing [20,21]. Platelet-shaped hexaferrites may be suitable as microwave absorption coating [29]. In the present study, the grains grew larger and became compactly stacked as the sintering temperature was raised. Obviously, the porosity and grain boundaries decrease with the rise of the sintering temperature [22]. The prepared nanocrystalline Sr<sub>2</sub>Y hexaferrite particles with larger grain size might be suitable as microwave absorbing materials.

### 3.6 Magnetic studies

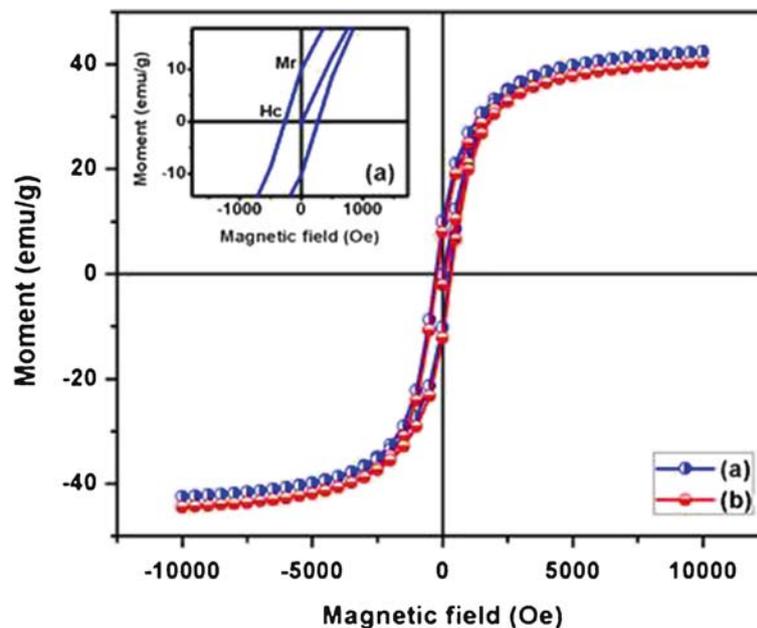
The magnetization of the present calcined Sr<sub>2</sub>Y hexaferrites was measured using vibrating sample magnetometer. Figure 7 shows plots of the individual hysteresis loops of the calcined B-Sr<sub>2</sub>Y and C-Sr<sub>2</sub>Y hexaferrites with applied magnetic fields up to 10 kOe. From these hysteresis loops, we can see that the saturation magnetization ( $M_s$ ) was high, while the coercivity ( $H_c$ ) was low. The values of saturation magnetization ( $M_s$ ), coercivity ( $H_c$ ) and remanence magnetization ( $M_r$ ) obtained from the hysteresis loops are listed in table 1. From the observed values of the coercivity ( $H_c$ ), it is clear that the calcined B-Sr<sub>2</sub>Y and C-Sr<sub>2</sub>Y show soft magnetic behaviour. It has been reported



**Figure 6.** FESEM images of C-Sr<sub>2</sub>Y hexaferrite nanoparticles at 1000°C with different magnifications.

earlier that the observed soft magnetic behaviour in ferrites is very significant for various practical applications in the fields of sensing, switching and security [23,24].

The room-temperature saturation magnetization ( $M_s$ ) of the calcined B-Sr<sub>2</sub>Y and C-Sr<sub>2</sub>Y nanocrystalline hexaferrite particles was found to be 42.44 and 38.72 emu g<sup>-1</sup> respectively. Similar results have been



**Figure 7.**  $M$ - $H$  loop of (a) B-Sr<sub>2</sub>Y hexaferrite nanoparticles at 1100°C and (b) C-Sr<sub>2</sub>Y hexaferrite nanoparticles at 1000°C with applied magnetic field up to 10 kOe. Inset figure shows the magnified view of B-Sr<sub>2</sub>Y at 1100°C.

**Table 1.** Magnetic parameters obtained from synthesized Sr<sub>2</sub>Y hexaferrite nanoparticles in comparison with other Y-type hexaferrites.

Y-type hexaferrites	Synthesis	$H_c$ (kA m <sup>-1</sup> )	$M_s$ (emu g <sup>-1</sup> )	$M_r$ (emu g <sup>-1</sup> )	SQR	Ref.
Ni <sub>2</sub> Y	Gel to crystallite conversion method at 950°C	16	25.5	–	–	[24]
Mg <sub>2</sub> Y	–	–	23	–	–	[25]
Zn <sub>2</sub> Y	–	–	42	–	–	[25]
Mn <sub>2</sub> Y	–	–	31	–	–	[25]
Zn <sub>1.2</sub> Cu <sub>0.8</sub> Y	1050°C	6.8	34.5	4.05	–	[27]
Co <sub>2</sub> Y	–	4.4	34	–	–	[25,29]
	Sol–gel derived fibres, 1000°C	30	32.8	–	–	[30]
	EDTA complex synthesis 1000°C	15–6.7	28.8–33.4	5.3–14.4	–	[31]
	1000°C	12.7	26.6	–	–	[32,33]
Sr <sub>2</sub> Y	Combustion method at 1100°C	21.33	42.44	10.05	0.24	This work
	1000°C	19.66	38.72	13.19	0.34	

reported for various Y-type hexaferrites [25–27]. Generally, differences in  $M_s$  depend on the temperature of calcination and particle size [28]. In our work, it is observed that the saturation magnetization slightly decreased by varying the fuel stoichiometric ratio conditions and the calcining temperature. The values of coercivity ( $H_c$ ) of the calcined B-Sr<sub>2</sub>Y and C-Sr<sub>2</sub>Y hexaferrites were found to be 21.33 and 19.66 kA m<sup>-1</sup> which are in good agreement with various values for Y-type hexaferrites reported by other groups [29–33]. The values of remanence magnetization or retentivity ( $M_r$ ) extracted from the  $M$ – $H$  loop were found to be 10.05, 13.19 emu g<sup>-1</sup> for the calcined B-Sr<sub>2</sub>Y and C-Sr<sub>2</sub>Y nanocrystalline hexaferrite particles. The squareness ratios (SQR) were calculated from the values of  $M_s$  and  $M_r$  and found to be 0.24 and 0.34 for the calcined B-Sr<sub>2</sub>Y and C-Sr<sub>2</sub>Y hexaferrites respectively. In general, large SQR values are preferred for many applications such as magnetic recording media and permanent magnets. The magnetic parameters of the prepared Sr<sub>2</sub>Y samples in this study are not only correlated with the grain size but also with the preparation conditions. It is possible to obtain higher quality samples with desirable magnetic properties by optimizing the processing parameters of the preparation.

#### 4. Conclusion

A new compound of Sr-based Y-type hexaferrite was successfully synthesized by autocombustion using glycine as the fuel. The newly prepared nanocrystalline Sr<sub>2</sub>Y hexaferrite particles were characterized by

XRD, FESEM, FTIR, XPS and VSM. The formation of a single phase with a nominal chemical composition of Sr<sub>2</sub>Mg<sub>2</sub>Fe<sub>12</sub>O<sub>22</sub> hexaferrites was demonstrated. The prepared Sr<sub>2</sub>Y hexaferrites showed good magnetic properties similar to that of the Y-type hexaferrites. The preparation of nanocrystalline Sr<sub>2</sub>Y hexaferrites particles depends on the initial conditions of the auto-combustion and calcination treatments.

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