

# Synthesis, Characterization and DC conductivity of Barium Hexaferrite

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**Abstract**— Barium hexaferrite were synthesized for three different Fe/Ba molar ratios 11:1, 11:2 and 11:3. All the samples were characterized by XRD for structural studies, FTIR spectra to confirm the formation barium hexaferrites and the SEM image of barium hexaferrite for understanding the surface morphology of barium hexaferrite. The DC conductivity of barium hexaferrite of all compositions shows almost similar electrical resistance and higher curie temperature & activation energy of these hexaferrites lies in  $7.13-12.1 \times 10^{-5}$  eV.

**Key words:** Barium hexaferrite, X-ray diffraction, FTIR, SEM, Dc conductivity.

## 1 INTRODUCTION

FERRITES Ferrites form a very good class of electrical materials because of their high resistivity and low loss behaviour, and hence have vast technological applications over a wide range of frequencies. Ferrites are preferred in the field of electronics and telecommunication industry because of their novel electrical properties which makes them useful in radio-frequency circuits, high quality filters, rod antennas, transformer cores, read/write heads for high digital tapes and other devices. Hence it is important to study their dielectric behaviour at different frequencies. The dielectric properties of ferrites are dependent on several factors, such as method of preparation, heat treatment, sintering conditions, chemical composition, cation distribution and crystallite size [1].

Ferrites, a distinct class of magnetic materials known as ferromagnetic have spinel structure. They consist of spontaneously magnetized domains and show the phenomena of magnetic saturation and hysteresis. Spinel ferrites possess properties of both magnetic materials and insulators and are important in many technological applications. The interesting physical and magnetic properties of spinel ferrites arise from the ability of these compounds to distribute the cations among the available tetrahedral (A) and octahedral (B) sites [2]. Spinel ferrites have gained lot of attention because of their remarkably high electrical and magnetic flux induction. They are considered as good dielectric and are found in many technological applications. Increased application of ferrites has led to the

hydrothermal, co-precipitation and sol-gel for the preparation of stoichiometric and chemically pure spinel ferrites [3].

Ferrites having low resistivity and low eddy current losses have been found to be the most versatile to be used for technological applications as in the case of stress sensors and recording media. Calcium ferrite possesses an inverse spinel structure and the degree of inversion depends upon the heat treatment [4], [5]. The physical properties of the spinel ferrite developed due to distribution of cations among the tetrahedral A site and octahedral B sites. The dielectric properties and conductivity of the ferrites depend on preparation method, chemical composition and grain size, frequency and temperature [6], [7]. DC resistivity of ferrites as a function of temperature and drift mobility and activation energy was reported by different authors [8], [9], [10]. Several researchers reported the magnetic properties of spinel ferrites as well [11], [12]. Ferrites is low mobility semiconductors. To know about the conduction mechanism in ferrites thermoelectric measurement is done. The thermo e.m.f. and its sign gives appropriate information about the type of conduction in semiconductors, i.e., they are p-type or n-type. There has been considerable interest during the past 10 years in finding new materials and structures for use in clear, highly efficient cooling and energy conversion systems [13] – [19].

## 2 EXPERIMENTAL

### 2.1 Synthesis of barium hexferrite powder

The starting materials used were barium chloride ( $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ ), iron chloride ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ), sodium hydroxide (NaOH) and sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) of Thomas Baker, Himedia and s. d. fine chemicals.

Barium hexaferrite were synthesized for three different Fe/Ba molar ratios 11:1, 11:2 and 11:3. Barium and iron chloride was

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development of many chemical methods which includes

weighed in stoichiometric amount and dissolved in distilled water. After complete dissolution of chlorides and NaOH/Na<sub>2</sub>CO<sub>3</sub> to form respective solutions; the NaOH/Na<sub>2</sub>CO<sub>3</sub> base solution was added drop by drop into Fe-Ba chloride solution with constant stirring till the pH of the solution became 11. The formation of complex starts precipitation and the precipitate obtained at pH 11 was washed several times with distilled water in order to remove the traces of NaCl from the precipitate. The precipitate was dried and presintered at 400°C for 2 hours.

The presintered precipitate was milled into fine powder for ½ hours in pestle and mortar. Polyvinyl alcohol solution in distilled water was mixed to the powder along with constant milling. PVA solution was used as binder for pelletization. The powder to PVA weight percent ratio was 20:1. PVA mixed powder was pressed into tablets by hydraulic press applying pressure of 10 tons. The pellets were sintered at 750°C, in a muffle furnace under air atmosphere for 6 hours. For temperature dependent conductivity, the pellets are coated with silver paste on either side of the surfaces. The copper electrodes are placed on each of the surface to obtain better contacts.

The IR spectra of the samples are recorded on Perkin-Elmer (Model 783) IR spectrometer in KBr medium at room temperature. For recording IR spectra, powders are mixed with KBr in the ratio 1:25 by weight to ensure uniform dispersion in KBr pellets. The mixed powders are pressed in a cylindrical die to obtain clean discs of approximately 1mm thickness. The percentage transmittances for the entire sample are measured from 300 to 4000 cm<sup>-1</sup>.

The X-ray diffraction (XRD) pattern of the BaFe<sub>12</sub>O<sub>19</sub> was recorded at room temperature by employing an x-ray powder diffractometer (Rigaku Miniflex) with CuK<sub>α</sub> radiation (λ=1.5405Å) in the 2λ(Bragg angles) range (2° ≤ 2θ ≤ 10°) at a scan speed of 0.5° minute<sup>-1</sup>. Optical micrographs of the films were made through a polarizing microscope (Nikon Eclipse E 400 POL) equipped with a digital camera.

The SEM images of polyaniline cadmium oxide composites were recorded using Philips XL-30 (ESEM) scanning electron microscopy. An analysis of the DC conductivity properties of the BaFe<sub>12</sub>O<sub>19</sub> has been carried out using Kethiely meter by two probe method.

### 3 RESULTS AND DISCUSSION

#### 3.1 X-ray diffraction studies

The X ray diffraction patterns of barium hexaferrite powder sintered at 750°C, is shown in figure. It revealed the formation of polycrystalline magnetoplumbite structure with preferred texture along (114). The traces of intermediate phases such as γ-Fe<sub>2</sub>O<sub>3</sub> or BaC<sub>2</sub>.2H<sub>2</sub>O were not observed in the X ray diffraction pattern. The observed 'd' values were in good agreement

with the standard 'd' values given in JCPDS data (84-0757). All the obtained XRD peaks were indexed with (hkl) planes from the mentioned JCPDS file which revealed formation of pure single hexagonal crystal phase.

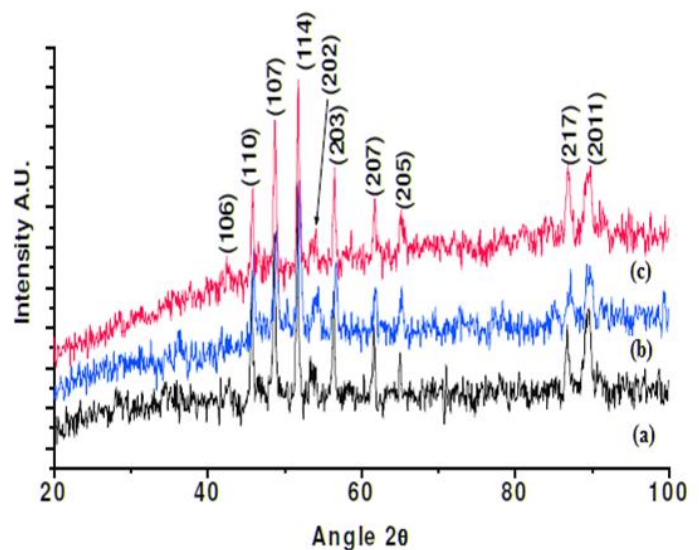


Figure 1 (a-c) shows the XRD spectra of barium hexaferrites (Fe/Ba ratio is 11:1, 11:2 and 11:3) sintered at 750°C.

To study the doping effect of Ba<sup>2+</sup> ions on crystal structure, barium hexaferrite powder of various weight percentage (Fe/Ba ratio is 11:1, 11:2 and 11:3) were sintered at 750°C is shown in figure 1(a-c). For all Fe/Ba ratios, the increase in Ba<sup>2+</sup> ions concentration in ferrites increases intensity of peaks due to increase in crystallinity of the material as expected. No impurity peaks were observed. Crystallite size of 11:1, 11:2 and 11:3 (Fe/Ba) weight percentage barium hexaferrite calculated by Debye Scherrer's formula is found to be 26.23 nm, 33.24 nm and 37.86 nm respectively.

#### 3.2 Fourier Infrared spectroscopy

The FTIR spectrum of barium doped hexaferrite (Fe/Ba ratio is 11:1, 11:2 and 11:3) sintered at 750°C is shown in figure 2 (a-c). All the FTIR spectra show three signature absorption peaks of hexaferrite [20] at ~ 436 cm<sup>-1</sup>, 546cm<sup>-1</sup> and 584cm<sup>-1</sup>. The peak at 436 cm<sup>-1</sup> indicate A<sub>2u</sub> vibration of octahedral Fe<sup>(4+)</sup>-O bonds and other two peaks indicate E<sub>1u</sub> vibration of Fe<sup>(3+)</sup>O<sub>4</sub> octahedra [21]. In case of barium hexaferrite prepared at the ration of 11:3, the absorption at 549 cm<sup>-1</sup> becomes higher than that of barium hexaferrite prepared at the ration of 11:1 and 11:2. The peaks at 436cm<sup>-1</sup> and 584cm<sup>-1</sup> are intense showing almost 60% absorption whereas a small notch at 546 cm<sup>-1</sup> was observed for all barium hexaferrite.

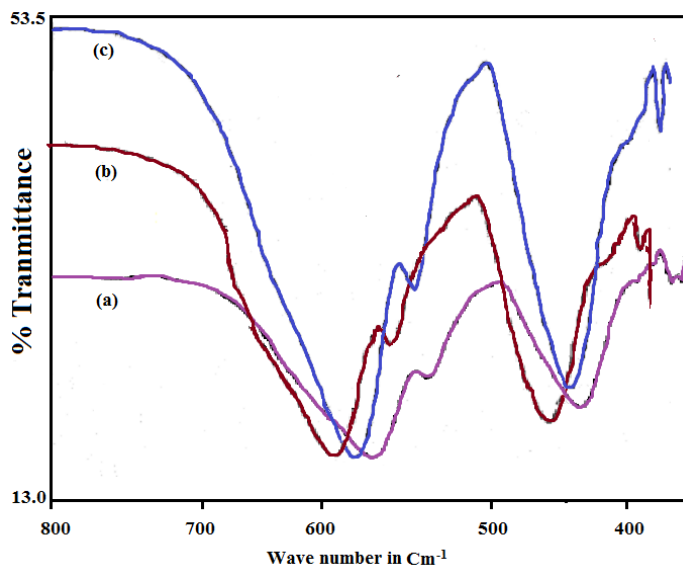


Figure 2 (a-c) shows the FTIR spectra of barium hexaferrites (Fe/Ba ratio is 11:1, 11:2 and 11:3) sintered at 750°C.

### 3.3 Scanning Electron Microscopy

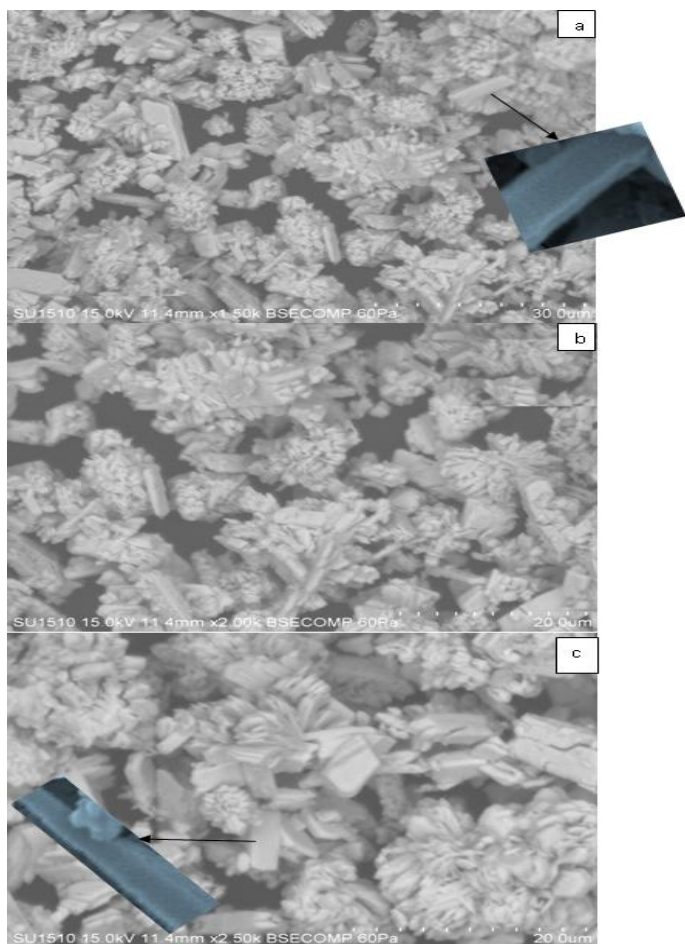


Figure 3(a-c) shows the SEM image of barium hexaferrites (Fe/Ba ratio is 11:1, 11:2 and 11:3) sintered at 750°C.

The SEM images of 11:1, 11:2 and 11:3 barium hexaferrite bulk are shown in figure 3(a-c). The SEM image of pH11 of 11:1, 11:2 and 11:3 barium hexaferrite bulk pellets sintered at 750°C showed various shaped grains as rectangular plates, nanorods and nanoneedles. The nanorods and nanoneedles like structure was observed at some places but degree of their formation was not enough to elaborate. The SEM image of barium hexaferrite (Fe/Ba ratio 11:1 & 11:2) at 750°C showed large number of vertically grown barium hexaferrite nanorods developed at some places over the rectangular platelets. The rods having length ~1000nm and diameter ~80nm were developed on the surface of dense rectangular grains.

The surface morphology of 11:3 barium hexaferrite pellet revealed formation of dense uniform tubular grains as depicted and comparatively more number of vertically grown long barium hexaferrite nano-needles having length ~120 nm and base to tip average diameter ~50 nm developed on porous surface of plate like grains of size ~100nm. The magnified SEM images of nanorod and nanoneedle obtained from scanning electron microscope is shown in inset.

### 3.4 DC conductivity

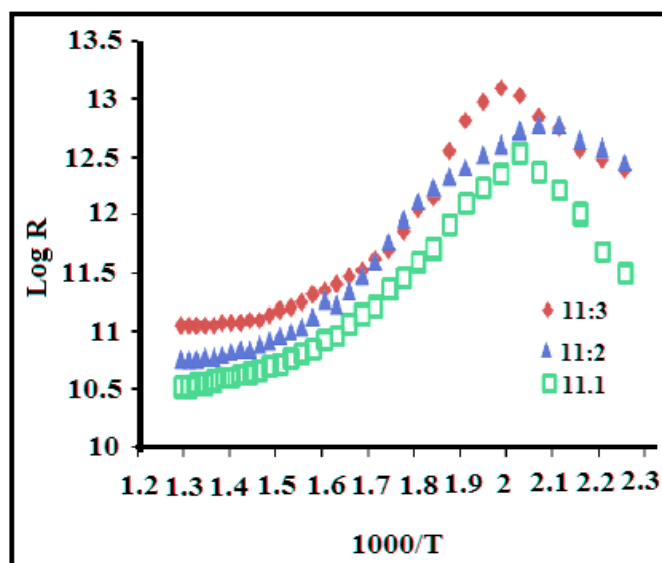


Figure 4(a-c) shows the DC conductivity of barium hexaferrites (Fe/Ba ratio is 11:1, 11:2 and 11:3) sintered at 750°C.

Figure 4 (a-c) shows variation of electrical resistance of barium hexaferrite (Fe/Ba ratio is 11:1, 11:2 and 11:3) pellets of diameter 1 cm and thickness 0.5cm with temperature. All barium hexaferrite show almost similar electrical resistance and higher curie temperature. The activation energy of these hexaferrites was calculated by well known Nernst Einstein relation [22]. The activation energy these hexaferrites lies in  $7.13-12.1 \times 10^{-5}$  eV.

The barium hexaferrite of all Fe/Ba ratios shows high resistance. Unlike bulk barium hexaferrite, the difference in curie temperatures was not significant for all. The Curie temperature and activation energy of barium hexaferrite is tabulated in table 1.

Samples	Curie Temp (°K)	Ea x 10 <sup>-5</sup> eV
11:1 BaFe12O19 bulk	280.00	7.17
11:2 BaFe12O19 bulk	250.00	6.46
11:3 BaFe12O19 bulk	210.00	5.43

#### 4 CONCLUSION

Barium hexaferrite were synthesized for three different Fe/Ba molar ratios 11:1, 11:2 and 11:3. From XRD spectra, is found that for all Fe/Ba ratios, the increase in Ba<sup>+</sup> ions concentration in ferrites increases intensity of peaks due to increase in crystallinity of the material as expected. The barium hexaferrite calculated by Debye Scherrer's formula is found to be 26.23 nm, 33.24 nm and 37.86 nm respectively. The FTIR spectra show three important signature absorption peaks of hexaferrite at ~ 436 cm<sup>-1</sup>, 546cm<sup>-1</sup> and 584cm<sup>-1</sup>. These peaks confirm the formation barium hexaferrites. The SEM image of barium hexaferrite (Fe/Ba ratio 11:1 & 11:2) at 750°C showed large number of vertically grown barium hexaferrite nanorods developed at some places over the rectangular platelets. The rods having length ~1000nm and diameter ~80nm were developed on the surface of dense rectangular grains. The surface morphology of 11:3 barium hexaferrite pellet revealed formation of dense uniform tubular grains as depicted and comparatively more number of vertically grown long barium hexaferrite nano-needles having length ~120 nm and base to tip average diameter ~50 nm developed on porous surface of plate like grains of size ~100nm. DC conductivity of barium hexaferrite of all compositions shows almost similar electrical resistance and higher Curie temperature. The activation energy of these hexaferrites lies in 7.13-12.1 ×10<sup>-5</sup> eV.

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