Photoluminescence behaviour of Gd doped barium hexaferrite

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Abstract. This paper reports synthesis of Barium Hexaferrite with different concentration of Gadolinium ion (0-2 mol %). Structural analysis of the prepared powders was done by X-ray diffraction pattern which reveals formation of hexagonal structure with space group p6/mmc. The FTIR and EDX spectra depict formation and composition of Barium Hexaferrite respectively. Agglomeration was observed from the SEM image of the prepared sample. Photoluminescence studies of the prepared samples show intensified PL emission peaks at 658 nm under ultraviolet (UV) excitation at 230 nm due to the optical transition 6G7/2 to 6P5/2 which arises because of the defects produced by doping of Gadolinium ion into the host lattice. Due to cross relaxation process PL intensity decreases after 1.5 mol% of doping concentration. CIE (commission International de) coordinates and CCT (correlated colour temperature) were also determined for the prepared samples which indicate that the powder samples show intense red cool emission.

1. Introduction
Hexaferrites having general formula MFe_{12}O_{19} where (M=Pb, Sr, Ba etc) were used in microwave devices, magnetic recording media, permanent magnetic materials because of their outstanding properties like high coercivity, high saturation magnetization, high curie temperature and good chemical stability and large uniaxial magnetic anisotropy [1]. Depending upon the crystal structure Hexaferrites were classified into 6 types such as X-type, W-type, M-type, Z-type and U-type [2].

Barium Hexaferrite (BaM) belongs to M-type hexaferrite with magnetoplumbite structure. Crystalline BaM particles have space group p6/mmc. The unit cell of the barium hexaferrite consists of 64 ions which are distributed in 11 different symmetric sites. Fe^{3+} ions are distributed in 1 trigonal bipyramidal (2b), 1 tetrahedral (4f1), and 3 octahedral (12k, 2a, 4f2) sites. Barium Hexaferrites has been studied by a large number of researchers worldwide because of their superior magnetic properties and excellent electrical properties like low eddy current losses and high resistivity [3].

Rare earth (RE) activated materials now a day’s grabbing attention because of their enhanced optical properties such as greater radiation stability, energy output, colour rendering index and enhanced luminescence intensity [4].

Keeping in view, the synthesis of Gadolinium doped Barium Hexaferrite by solution combustion technique and it’s structural, morphological and photoluminescence behaviour has been reported in this paper.
2. Material and Synthesis
Barium Hexaferrite samples doped with different concentration of Gadolinium were synthesized by solution combustion technique. Barium nitrate, Ferric nitrate and Gadolinium nitrate all are supplied by sigma Aldrich with more than 99% purity were taken in appropriate stoichiometric amount and dissolved in deionized water. Further, the solution was placed on magnetic stirrer and citric acid as fuel added drop wise with continuous stirring. When temperature of the magnetic stirrer reaches 80°C the self ignition process starts and we get the nano porous powder. The porous powder was ground finely and placed in a high temperature furnace for calcination at 1200°C for 3 hour.

3. Experimental Results and Discussion
3.1 Structural Analysis
The crystallographic characteristics of the synthesized powder samples were determined by the XRD pattern obtained from the PAN Analytical X-Pert Powder Diffractometer with Cu-K alpha radiation ($\lambda=1.54\text{Å}$). Figure 1 represents XRD pattern of undoped and Gd$^{3+}$ ions doped Barium Hexaferrite which confirms that the prepared samples have hexagonal structure with space group p6$_3$/mmc. The average particle size of the prepared samples calculated from Scherrer equation is 28.17nm.

![Figure 1. XRD pattern of undoped and Gd doped (1.5mol %) Barium Hexaferrite](image)

3.2 Morphological Analysis
Figure 2 shows SEM image of prepared sample BaFe$_{12-x}$Gd$_x$O$_{19}$ (x=1.5mol %) which was taken by ZEISS Ultraplus SEM Analyzer. It is clear that the particles were agglomerated with irregular shape [5].
3.3 Elemental Analysis

EDXS of the prepared sample is as shown in figure 3. The intense peak corresponding to Oxygen (O), Iron (Fe), Barium (Ba) and Gadolinium (Gd) confirms that all the starting materials are present in the prepared sample. The atomic and weight percentage of the starting materials are given in table 1.

Table 1. EDX Spectrum analysis data of the prepared sample.

| Element | Weight% | Atomic % |
|---------|---------|----------|
| O       | 31.38   | 65.48    |
| Fe      | 50.30   | 30.07    |
| Ba      | 18.03   | 4.38     |
| Gd      | 0.29    | 0.06     |

Figure 3. EDX Spectrum of the prepared sample.
3.4 FTIR Analysis
FTIR analysis has been performed to confirm the formation of phase of the prepared samples. Figure 4 represents FTIR spectra of the prepared samples which were recorded by Bruker FTIR spectrometer. The band corresponds to 500 cm\(^{-1}\) to 400 cm\(^{-1}\) confirms the formation of Barium Hexaferrite [6].

![Figure 4. FTIR Spectra of the prepared samples.](image)

3.5 Photoluminescence (PL) Studies
Photoluminescence emission spectra of Gd\(^{3+}\)ions doped Barium Hexaferrite samples were recorded by Shimadzu RF-5301PC spectrofluorophotometer. Figure 5 represents PL emission spectra of Gadolinium doped Barium Hexaferrites under UV excitation of 230nm. The most intense peaks were observed at 658nm due to the optical transition \(^6\)G\(_{7/2}\) to \(^6\)P\(_{5/2}\) which were originated from the defects level of Gd\(^{3+}\) ion. From the figure it is clear that with increasing the concentration of Gd\(^{3+}\) impurity upto 1.5 mol %, the intensity of the PL emission increases after that PL emission intensity gradually diminishes because of the cross relaxation process which arises from the non radiative transition between the dopant ions[7].
3.6 Chromaticity Characterization

The CIE (Commission International de) coordinates and CCT (correlated colour temperature) of 1.5 mol% Gd$^{3+}$ ions doped barium hexaferrite for which the PL intensity is maximum is shown in the table 2 to determine the accurate colour and nature of the prepared sample [8].

| Sample         | Concentration of Gd$^{3+}$ | X coordinate | Y coordinate | CCT(K) |
|----------------|----------------------------|--------------|--------------|--------|
| BaFe$_{12}$O$_{19}$:Gd$^{3+}$ | 1.5 mol %                  | 0.730        | 0.270        | 4605   |

It is clear from the figure that the sample shows deep red emission and the evaluated CCT value indicates that the prepared sample belongs to cool emission.

Figure 5. PL emission spectra of the prepared samples

Figure 6. CIE Diagram of 1.5 mol% Gd doped Barium Hexaferrite.
4. Conclusion

Barium Hexaferrites doped with Gd$^{3+}$ ions were prepared by solution combustion technique. Particles of the prepared samples have hexagonal structure with space group p63/mmc as confirmed by XRD pattern analysis. Gd$^{3+}$ ions were successfully incorporated into the host lattice as confirmed by EDX analysis. Photoluminescence spectroscopic studies reveal that BaFe$_{12-x}$Gd$_x$O$_{19}$(x=0 to 2 mol %) show PL emission spectra at 658nm corresponding to $^6$G$_{7/2}$ to $^4$P$_{5/2}$ optical transition of gadolinium ion. The chromaticity investigations confirm that the prepared samples can be applied to various display devices because of their cool red emission.

References

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