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Magnetic and Mössbauer studies of low temperature crystallined small size barium hexa ferrite nanoparticles

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Abstract : *Barium Hexa ferrite nanoparticles were synthesized using chemical based citrate precursor method. The Citrate precursor was annealed at temperatures 700°C, 750°C and 800°C in a muffle furnace that lead to ferrite powder after crushing. The powder samples were characterized using X-ray diffraction* ***(XRD),*** *Vibrating sample magnetometer (**VSM**), Scanning electron microscopy (**SEM**) and* ***Mössbauer spectroscopy****. The average particle size was observed 51nm, 89 nm and 44nm respectively for the above mentioned samples prepared at different annealing temperatures. The largest coercivity 4060 Oe was observed for sample annealed at 750°C. All the magnetic parameters were found to increase with annealing temperature upto 750°C and again decreased at 800°C. Mössbauer studies show that samples are in single phase with different magnetic characteristic i.e. magnetically coupled phase.*

Key words*: Hexa-ferrite, Nanoparticles, Low temperature, Mössbauer & Magnetic studies.*

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Introduction:

Barium hexa ferrite having general formula $BaFe_{12}O_{19}$ is a famous magnetic material which has a variety of applications in microwave devices and in permanent magnet *(Rodic et. al, 1999 and Chikazumi Soshin, 1996)*. The electromagnetic wave absorption property of hexaferrite with a magnetoplumbite structure in the GHz range, which includes M-, W-, Y-, Z-, U- and X-type has found demands for microwave communication, microwave dark room target camouflage, electromagnetic radiation abatement, and so on *(Carp. 1998)*. In nanocrystalline form hexaferrites are of particular interest for use as high density perpendicular magnetic recording media *(Dobrzanski et. al, 2006 and Ziebowicz et. al, 2007)*.

Many processes have been used by several researchers to prepare hexa ferrite, including the solid state method, Sol-gel and coprecipitation and other chemical processes *(Oikonomov et. al, 2007; Gil Kim Soon, et. al, 2007 ; Sugimoto Satoshi, 2005* ; *Mu Guohong et. al, 2007 and Singh et. al, 2010)*. Among these processes, coprecipitation and sol-gel methods are more popular for the preparation of hard ferrite nanomaterials. Recently, the nanocrystalline Ba hexa ferrite with average particle size below 100nm has been prepared by sol-gel process *(Sugimoto Satoshi, et. al, 2005)*. However such a small particle size is still difficult to achieve by simple coprecipitation method. **We have prepared small particle of size of 51nm, 89nm and 44nm (less than 100nm) at annealing temperature, 700°C, 750°C and 800°C using Citrate precursor method. This may be a special feature of this chemical method**.

Materials and Methods: Laboratory Preparation

Samples of nanometer-sized Barium hexaferrite powder were prepared by using the Citrate precursor method. Ferric nitrate, Barium nitrate and Citric acid were taken in Stoichiometric proportion as starting materials. Aqueous solutions of these salts were prepared separately by dissolving the salt in minimum amount of deionized water while stirring constantly. The solutions were then mixed together. The mixture was heated to temperature between 60°C to 80°C for two hours with continuous stirring. This solution was allowed to cool to room temperature and finally it was dried over night in oven in order to remove excess water and other impurities at 90-95°C until it formed a brown color fluffy powder. The Citrate precursor was heated at temperatures 700°C, 750°C & 800°C for one hour in a muffle furnace. By this process, the precursor thermally decomposed to give Barium hexaferrite powder of nanometer size.

Results and Discussion:

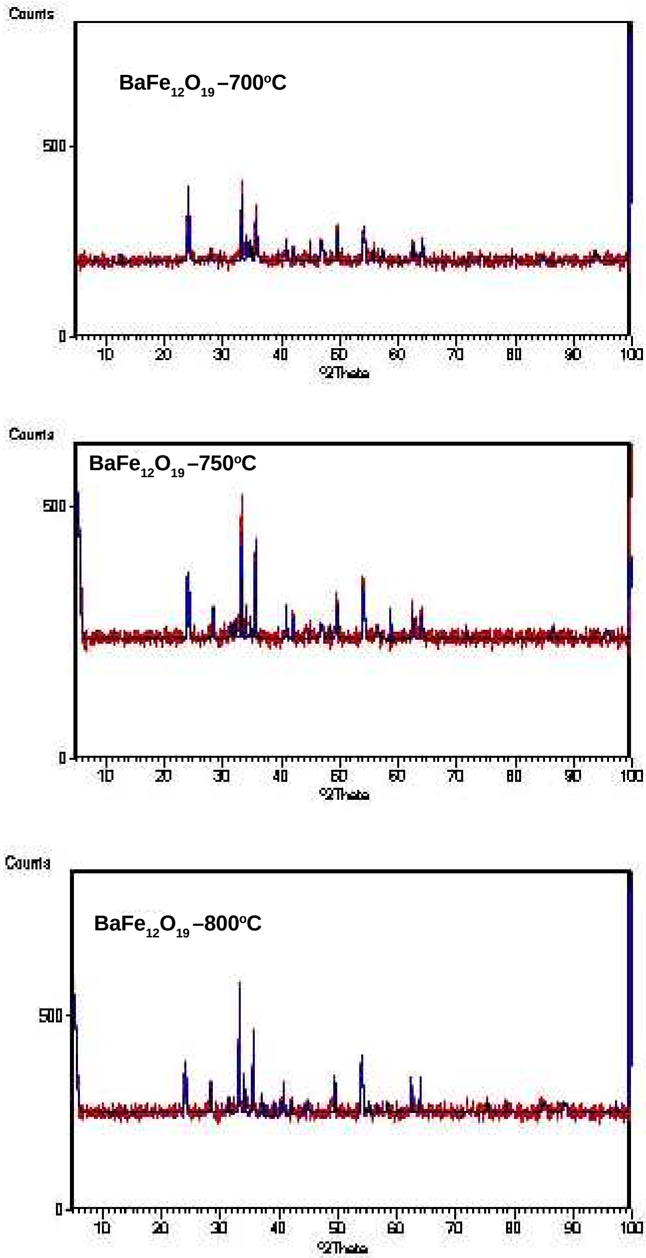

Figure1 : Xray Diffraction Patterns for $BaFe_{12}O_{19}$ Nanoparticles annealed at temperature 700°C, 750°C and 800°C

The X-ray diffraction spectra of synthesized materials is shown in figures 1. We have chosen maximum intensity peak for particle size calculation and particle size observed was 51 nm, 89nm and 44 nm at 700°C, 750°C & 800°C respectively using Scherrer formula *(Culity, 1978)*. All samples show Hexagonal structure as compared to JCPDS data base (Card. No-190098 and 78-0135) with some impurity.

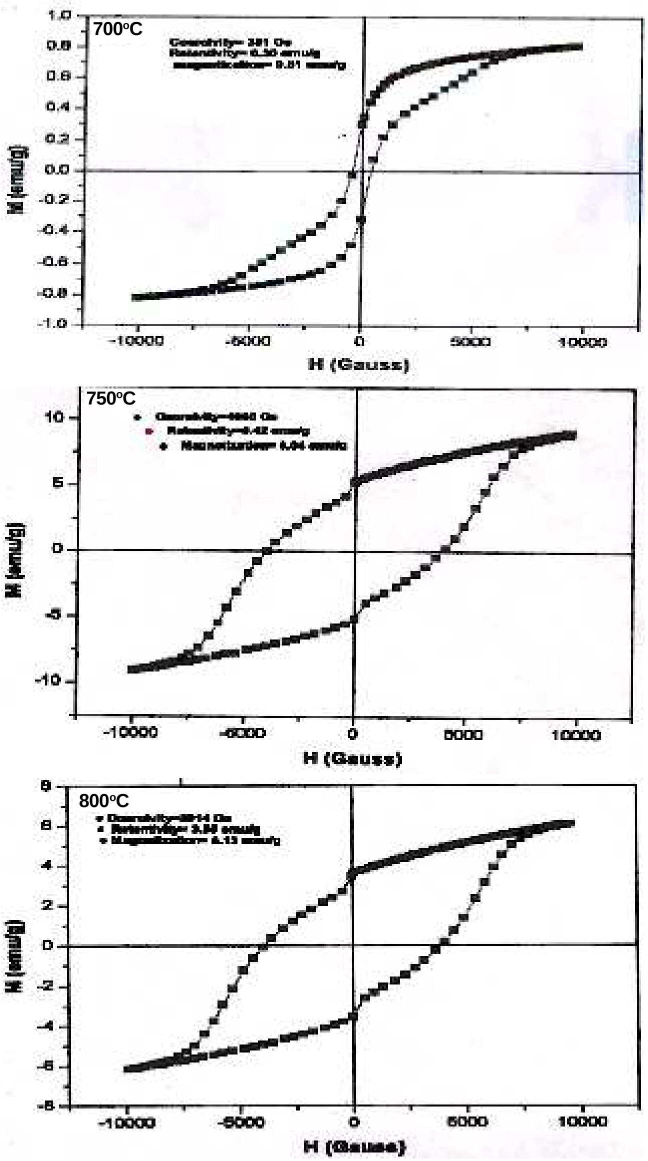

Figure 2: magnetization Curve for $BaFe_{12}O_{19}$ nanomaterials annealed at temperature 700°C, 750°C and 800°C

This impurity may be due to some residual unreacted part. The SEM image and Mössbauer spectrum and are shown in Figure 3 and Figure 4.

Table 1: Observed data for Ba Hexa Ferrite ferrite nanoparticles.

Annealing temperature	Mean particle size	Coercivity H_c (Oe)	Retentivity M_r (emu/g)	Saturation magnetization M_s(emu/g)
700°C	51 nm	381 Oe	0.30 emu/g	0.81 emu/g
750°C	89 nm	4060 Oe	5.42 emu/g	9.04 emu/g
800°C	44 nm	3914 Oe	3.55 emu/g	6.13 emu/g

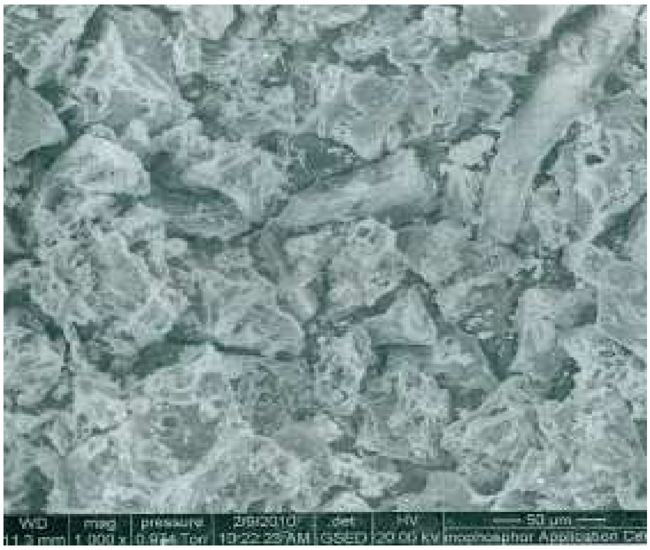

Figure 3 : Morphology of $BaFe_{12}O_{19}$ nanomaterials, annealed at 750°C

In Mössbauer spectrum (Fig. 4) relative intensity (T%) gives the information that the particles are randomly oriented and are in single phase. There are minimum two missing sextent from the Mossabuer spectrum one is near to 33.0 - 35.0T and the other one with more magnetic hyperfine interaction.

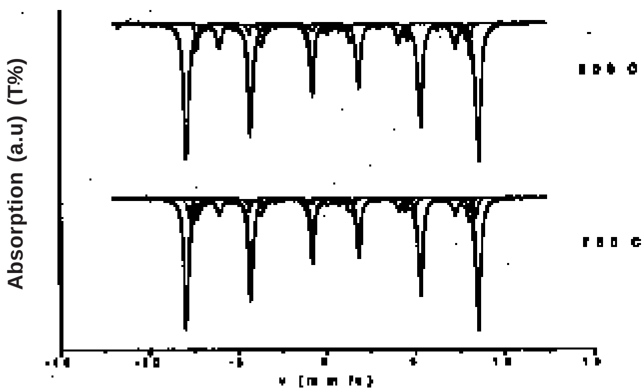

Figure 4 : Mössbauer spectrum of Barium Hexa ferrite Nanoparticles annealed at 750°C and 800°C

By looking at the graph one can easily say the phases present. They are magnetically coupled phases. Low value of width shows that each phase is singlephase having different magnetic characteristics. This result confirms the observation of high value of coercivity (4060 Oe and 3914 Oe) in the samples annealed at temperature 750°C, and 800°C. SEM image shows, cluster of fine particles and has a tendency to form larger and more spherical particles.

The magnetization curve obtained from VSM are shown in figure 2 (Magnetization & Field are along Y and X axis and the magnetic parameters are tabulated in table 1.

In the preparation of hexaferrite, a high annealing temperature is required to obtain pure phase. This results in significant increase of particle size together with improvement of ion occupancy *(Kaczmarek et. al, 2004)*. It has been reported *(Wang Jun et. al, 2004)* that the Barium hexaferrite nanoparticles formed at 140°C in presence of 0.25T magnetic field exhibit a higher saturation magnetisation i.e. 6.1emu/g at room temperature as compared to 1.1 emu/g obtained for sample prepared in zero magnetic field. Few researcher *(Sankaranarayanan and Khan 1996)* have reported in their work that magnetization first decreases with increasing annealing temperature, reaches a minimum for annealing temperature (417°C), and then increases to reach a maximum for annealing temperature (447°C), before decreasing again sharply to approach zero. Several researchers found *(Kaczmarek et. al, 2004)* that annealing in air promotes slightly higher H_c value. They assume that different particle morphology, is directly responsible for fluctuations in magnetic parameters. The value of coercivity increases over six times and reaches a value 445.6 kA/m. This value is typical of chemically coprecipited fine hexa-ferrite powders, where perfect crystal structure assures a defect and stress-free spin arrangement with high magnetocrystalline anisotropy energy *(Kaczmarek et. al, 2004)*. In this work Ba-hexa ferrite nano materials annealed at 750°C having coercivity 4060 Oe may be used for technological applications in Electronics *(Glebin et. al, 2005)*.

Conclusion :

We have prepared small size particle of 51nm, 89nm and 44nm(less than 100nm) at annealing temperatures, 700 °C, 750 °C and 800°C using Citrate precursor method. The maximum coercivity was observed 4060 Oe and 3914Oe at annealing temperature 750°C and 800°C. The larger particle size have larger coercivity. The structural and magnetic properties depends on particle size and annealing temperature. The Mossbauer studies shows that phases present are magnetically coupled phase.

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