SYNTHESIS AND CHARACTERIZATION OF RARE EARTH PRASEODYMIUM DOPED BARIUM-HEXAFERRITE

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By

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CERTIFICATE

Thereby certify that the work which is being presentedin the Dissertation entitled **""SYNTHESIS AND CHARACTERIZATION OF RARE EARTH PRASEODYMIUM DOPED BARIUM-HEXAFERRRITE"**inpartialfulfillmentoftherequirementfortheawardofdegree of **Masters in Sciences** and submitted in Department of Science and Technology ,Lovely Professional University, Punjabis an authentic record of our own work carried out during period of Dissertation under the supervision of **Dr. Kawaljeet Singh**, **Assistant Professor**, Department of Science and Technology, Lovely Professional University, Punjab.

The matter presented in this dissertation has not been submitted by me anywhere for the award of any other degree or to any other institute.

Date: 24-11-2014   **KARAMVEER CHAHAL**

This is to certify that the above statement made by the candidate is correct to best of my knowledge.

Date: 24-11-2014   **Dr. KAWALJEET SINGH SAMRA**

Supervisor

Signature of Examiner

Acknowledgement

I would like to thank "**Dr. Kawaljeet Singh Samra** and **A.K Srivastava**" Without their guidance and help, this project could not have been completed. Then I would like to thank my university "Lovely Professional University" for giving me a chance to work on such a fantastic project and exploring my knowledge and also gaining some experience.

Then I would like to thank my parents for all their blessing and support in terms of money and every aspect. Also I would like to thank my senior "**Talwinder Kaur**" for being a constant support and being there whenever in need.

Introduction

Barium ferrites are general molecular formula ($BaFe_{12}O_{19}$). It is well known for its high permanent magnetic operation and good mechanical hardness [1-2]. Now a days in electronics age, it has attracted more attention in recent years due to its high coercivity, large magnetization saturation and superior chemical stability and more corrosion resistivity [3-4] .This type of material contains a structure similar to magneto-plumbite. It is more application to manufacture permanent magnet materials and it is used in microwave devices technology for absorption of em waves, magnetic recording media to store a large amount of data in form of memory devices, speaker, micro-oven, radar communication, [5-6] . This type of material is known as M-type hexaferrite ($MFe_{12}O_{19}$). Barium plays an important in these Materials because it is highly corrosion power & less reactive towards oxidation. This is why barium dope ding is more important to protect our materials by external effects of moisture air, water & other acidic reactivity which effects on iron rusting.

The magnetic properties like coercivity and magnetization saturation of hexagonal magnetic ferrites can be enhanced over cationic replacement of Fe_3 ions in the sub framework of the crystal. In this paper our main focus is to analyses the effects of doping of f-block elements with respect to temperature's-block elements are also known as rare earth elements, such as Presidium (Pr). Now general formula of M-hexaferrite changes to $Ba_{1-x}Pr_xCo_xFe_{12-x}O_{19}$.there are many synthesis method for the formation of hexaferrite materials such as sol gel, citrate precursor method, micro emulsion method, milling method and micro wave induction method. But in this paper we have studied the m-type hexaferrite

$Ba_{1-x}Pr_xCo_xFe_{12-x}O_{19}$ (BaPrCM) is manufactured by sol-gel auto burning method. The purpose of work is to study the effects of temperature consequence on physical orientation, magnetic properties, effects of heat on mass of sample etc. This type of ferrite materials is low cost and hence can be easily used by a simple personin many electronics things & commercially worldwide field of uses.

Rationale and Scope of Study:

The study of hexaferrite materials is extremely useful in field of electronics field and daily life due to its high permanent magnetic performance and good mechanical hardness. It is also used in microwave oven, speaker, electronics toys, in recent time's perpendicular and longitudinal larger density magnetic recording data of communications, Wi-Fi communications, memory devices etc.

Objectives of the study:

The basic objective of this project is to:

➢ Synthesis of Praseodymium rare doped Barium hexaferrite.

➢ Study about variation in the magnetic and electrical properties of synthesized material at different temperature.

➢ To learn the consequence of temperature while synthesis on operational properties of Praseodymium rare doped Barium hexaferrite

➢ To increase the storage capacity of the memory device.

Review of Literature:

In 2015, Talwinder kaur et.al:

The synthesized M-type hexaferrite ,$Ba_{0.7}La_{0.3}Fe_{11.7}Co_{0.3}O_{19}$ powder of sample, by using sol gel auto combustion technique, at different heat rate at 700,900,1100,and 1200°C.the x-ray powder (XRD) characterization shows that formation of absolute phase of M-type hexaferrite after that used (TGA) thermo gravimetric analysis which shows that the weight loss of sample powder becomes constant after 680°C and then FTIR characterization shows two absorption peaks at 432 and 586 cm^{-1} give the formation of M-type hexaferrite .the magnetic property analysis by VSM ,it shows that maximum value of coercively value (5602 Oe) is found at 900°c.the band gap of sample studied by UV-vis NIR spectroscopy ,it shows that pure sample BaM low value then BaLCM, it shows that doping increase band gap of sample.

In 2014, Guk-Hwan An et.al:

The synthesized M-type barium hexaferrite sample by salt- assisted ultrasonic spray pyrolysis at different range of temperature. NaCl salt is used added ,it melted at 850,900& 950°C to act like a solvent in the reaction .the particles characterized by XRD,SEM,& TEM analyses .the sample free from salt is obtained at 950°C ,it shown that existence of only barium hexaferrite single phase. After that salt is added barium hexaferrite sample is synthesized at 950°C.it shows hexagonal plate shape, with size of grain is 72nm & good crystallinity. The vsm studded, at room temperature. The sample of salt added shows coercively 5735 Oe & magnetic saturation value is 63,2 emu/g at 950 °C .

In 2013, Ihsan Ali et.al :

The synthesized M-type hexaferrite ($BaFe_{12}O_{19}$) sample powder by sol gel auto combustion method at heat of1000°C for different intervals (t=1,2,3& 4 h).the characterization DTG and DSC are agreed that the observe weight loss &conversion of phase variations all through heat analysis. An XRD arrangement gives the formation of only one phase of hexaferrite. Dielectric constant & value of dielectric loss decrease when ac supply conductivity increase with growing of frequency. The magnetic property like saturation of magnetization & coactivity improved

value range (4704-5070 Oe) with the increase of temperature. The improved property useful for high frequency application, perm ant magnet &recording media.

In 2013,GUOLONG TAN et.al :

The synthesized M-type hexaferrite material ($SrFe_{12}O_{19}$) by using co-precipitation precursor technique. The sample is sintered into ceramics at range of temperature 1000-1100°C for 1h.the characterization tool used for analysis morphology & structure by field emission scanning (FE-SEM) &XRD respectively .the remnant polarization maximum value found at temperature 1000°C is $15\mu C/cm^2$.

The remnant magnetic polarization & coactivity (Hc) of 34emu/g and 4180 Oe respectively at temperature range 1000°C.

In 2012, S.Kanagesanet.al :

The M type Barium hexaferrite ($Ba_{0.5}Sr_{0.5}Fe_{12}O_{19}$-BSF) synthesized by sol - gel technique used D-fructose such as a fuel. XRD-ray patterns shows of planes its structure like a hexagonal magneto plumbite (M-type) structure.HR-SEM measure the sample grains size with uniform shape spreading& hexagonal in form. The magnetic property analysis by vsm, the maximum saturation magnetization value 53.44 emu/greatest to grain size collection from 0.5-1,7μm.also found intrinsic coactivity (Hc) value 1998 gauss.

the remnant (Mr)magnetization & saturation (Ms)values are 42.64 &66.32 emu/g respectively .

In 2012, G.Asghar et.al:

Strontium hexaferrite manufactured by simple co-precipitation method.Xrd study, the only onstage of $MFe_{12}O_{19}$(M=Sr).the sample average particle size was found 18nm.as the frequency rise cross ponds to dielectric constant value decreases, because motion of charge carrier do not follow frequency of applied external field. The magnetic property, coercivity & magnetic saturation values are 2000 Oe& 44.3 emu/g. this value of coercivity is smaller than theoretically due to multi-domain grains.

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In 2011,M.M. Rashad et.al:

both Barium & strontium ,M-type hexaferrite are synthesized by novel approach co-prepetition approach method using 5M solution of sodium carbonate maintain pH=9 at different heat treatment from 800-1200 °Cat Fe^{+3}/Ba^{+2} molar proportion 12.the xrd study analysis that grain size of powder sample increased with raise in annealing temperature & molar ratio.vsm study shows that good value of saturation magnetization isMs=53.8 emu/g with coercivity (Hc)=4675 Oe was reached for clean single stage barium hexaferrite at range of temperature 800°C for 2h.other side magnetization saturation 85.4 emu/g with coercivity 4902 Oe was obtained for strontium hexaferrite pure state at Fe^{+3}/Sr^{+2} molar proportion value 12 & heated at 1000°C for 2h.the range of coercivity 3906 Oe of strontium ferrite powder is used for magnetic recording media application in floppy disk, hard disk ,video tape etc.

In 2011, Muhammad Javed Iqbal et.al:

M-type hexaferrite is important tool for electrical properties. This property of sample enhance by cationic substitution technique. The synthesized &description of Nd-Ni is drugged in strontium-barium hexaferrite of configuration

$Sr_{0.5}Ba_{0.5-x}Nd_xFe_{12-y}Ni_yFe_{12-y}Ni_yO_{19}$ value varies from(x = 0.0 - 0.1,y=0.0-1.0)by Co-precipitated method. The electrical properties are study using state- of –the- art method.Xrd analysis the lattice parameter & cell volume are reciprocal related to the doped material. This sample behaves same as semiconductor material above 350K such that resistivity decreases with raised a temperature. Resistivity value of undoped sample decay from a value of 1.8 x 10^{10} Ωcm to 2.0 x10^8Ωcm by the help of doped Nd-Ni ions.

In 2010, G.MurtazaRaiet.al :

In this paper doped of holmium in a M-type barium hexaferrite $BaFe_{12-2x}Ho_{2x}O_{19}$ with (x=0.0-1.0) .this sample is synthesized by solid state reaction method. Xrd analyses says that M-type structure exist with small few secondary phases also exist. The SEM studied that the grain size decreases with increasing concentration value of doped holmium. The holmium elements belong to rare earth elements HO^{+3} it acts as a grain size of growth inhibitor. The VSM analyses says that maximum value of coercivity of sample at (x=0.4) is 2230 Oe. Hence this sample used for application of high density storage devices.

In 2009, Mahmood*et.al.* :

Studied the M-type hexaferrite $BaFe_{12-x}Ga_xO_{19}$ sample powder with varying from(x= 0 to 1.0). The samples were developed by ball milling method and were analyses via Xrd, TEM, and VSM.Xrd study after analysis that the sample was establish that the elements and crystallites have comparable mean size of $\sim$41 nm for all analyzed sections. The saturation magnetization values of sample decreased slightly and nonlinearly with concentration of x increasing. The vsm studied that coercivity decreased to some extent for ($x\leq$0.2) concentrations of Ga doped content, and then further improved with increasing Ga value of x up to $x = 1.0$.

In 2009, Dube*et. al.*:

The synthesized of doped Zn-Ti M-type barium hexaferrite sample powder by solid state reaction method. The complex permittivity and permeability measurements of the sample of $BaFe_{12-2x}Zn_xTi_xO_{19}$ ($0.2\leq x\leq$0.6) were carried out by cavity perturbation technique in the microwave region of frequency range of 8–13 GHz. The LCR meter studied that abnormal dielectric behavior of sample, increasing value of dielectric constant with frequency was property of the mixed conduction state. The effect of doped Zn–Ti on the sample of the permittivity and dielectric loss was observed in the frequency collection of 1 kHz–1MHz. The increase concentration of Zn–Ti dopants in sample monotonically increased both the permittivity and the permeability in the observed frequency ranges, which were showed that increased polarizability and unpinning of the domain walls.

In 2010, Liu.*et al.* :

The M-type barium hexaferrite is synthesized doped with Co, Zn and Sn of the general formula $BaFe_{12-2x}Zn_{x/2}Co_{x/2}Sn_xO_{19}$ (x=0.0-2.0) by co precipitation technique. The characterization tool used for analyses XRD powder, VSM, ESEM/EDX. The results studied that the grain magnitude of the sample shrinkages with the increase in the doping x concentration of dopant materials. VSM studied, high saturation magnetization and coercivity was close to zero , when x range of doped sample between 0.3 and 0.4.

In 2007, Wang Lixi et.al:

The M-type barium hexaferrite is synthesized, doped with Sm^{+3} is general formula $Ba_{1-x}Sm_xFe_{12}O_{19}$ with variation(x=0.0,0.1) by conventional ceramic method. The structural & electromagnetic property studied by XRD powder & network analyzer. The XRD showed that the magnetoplumbite structure of a sample without other intermediate phase. The grain size decreased due to doped concentration of Sm^{+3}, resultant size of sample contracted. The study of microwave electromagnetic properties of powder sample at frequency range of 2 -18GHz.it showed that the dielectric constant values increase slightly with doped concentration of contents. Hence this sample is used to improve the absorbing performance of microwaves instruments.

Materials and Research Methodology:

Barium hexaferrite material doped with rare earth metal like praseodymium whose molecular formula is $Ba_{1-x} Pr_x Co_x Fe_{12-x} O_{19}$ is taken where x=0.4. Sol-gel technique for manufacturing of Barium hexaferrite materials is used.

In this process firstly molecular weight is taken in grams with the help of weighing machine and assumption is taken as metal: citric: 1:2. Diluted Barium solution is mixed with diluted praseodymium solution in a beaker and simultaneously cobalt and ferric nitrate solution and is mixed in another beaker. After obtaining the two solutions separately contents of beaker 1 are mixed with the contents of the beaker 2. After obtaining the solution; citric is added to the beaker as a fuel and stirred continuously using electromagnetic stirrer without heating. Then ammonia was added and a pH =6.50 is obtained using pH meter. Once the desired pH is obtained the solution is allowed to heat between 80-85°C until the formation of gel.

Once the whole solution has been converted into gel form, the magnetic stirrer is turned off and the beaker containing gel is kept on hot induction plate which has been maintained between the working ranges of 285-300° C so that the gel gets converted into powdered form. Once the gel is completely converted into powder form, it is allowed to be kept in a furnace by dividing the powder into four equal parts and each observed under different temperatures i.e. at temperature of 700°, 800°, 900°, 1000°for 4 hours each.

Characterization Tools:

For the study of sample of hexaferrite materials we used many characterization tools for different purpose of studying. For morphology of sample used XRD technics. The effects of heat w.r.t loss of masses analyzed by TGA/DSC by instruments. Proper bond formation of sample takes place is studied by FTIR. The magnetic property of sample studied by VSM instruments.

1. X-Ray Diffraction (XRD):

X-ray diffraction by crystals was detected in 1912 by Max von Laue.X-ray diffraction methods are the best methods for finding the crystal structure of materials.

X-ray like a em waves interact with sample of electron clouds due to its shortest wavelength. If incident X-ray beam on the sample of powder then some x-rays will be absorbed by the sample &other will be diffracted by the atoms of sample .It is based on diffraction patterns of principal.

X ray here shows particle as well as wave nature. We used Bragg's law for scattered beam of X-rays.

Bragg's diffraction law:

$$2d \, Sin(\theta)= n\lambda.$$

Here,

d=crystal plane separation between them.

λ=wavelength of X-ray.

n=order of constructive interference patterns.

θ=diffraction angle.

By this characterization we find structural parameters like average grain size, crystal defects, volume of cell , density, lattice plane ,miller indices of sample etc.

The particle size of sample calculated by Scherrer formula, using (FWHM)full width half maxima (β).

$$D=0.9\lambda/\beta \, Cos(\theta)$$

Here,

D=grain size of sample.

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β= full width half maxima of diffraction peak.

θ=diffraction angle.

For finding lattice constant value a and c respectively. We used this relation between lattice constant & miller indices of a plane.

$$\frac{1}{d_{hkl}^2} = \frac{4}{3}\left(\frac{h^2+hk+k^2}{a^2}\right) + \frac{l^2}{c^2}. [1]$$

Here,

    d=separation distance between crystal planes.

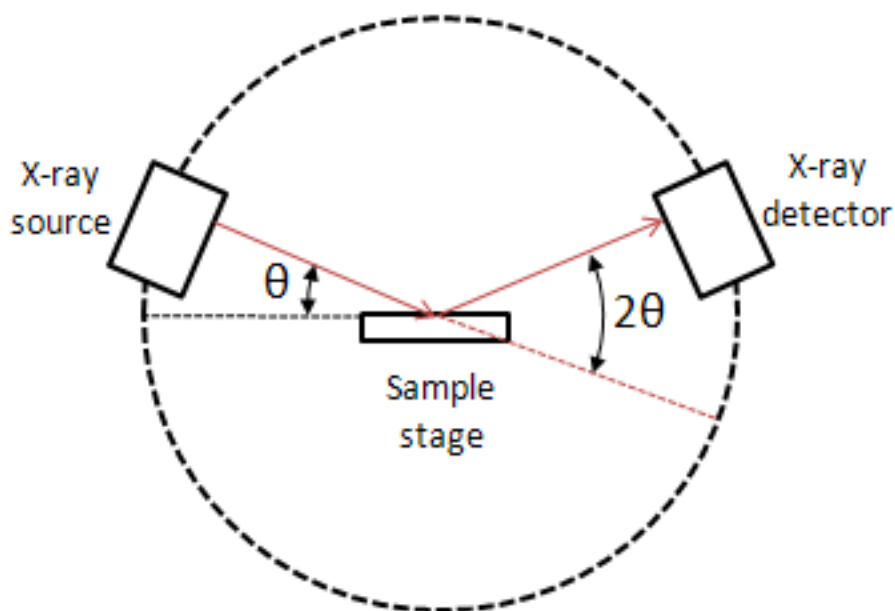h,k,l =miller indices of the crystal sample.

a&c=lattice constant parameter.

Fig: XRD powder diffraction schematic diagram.

Internet link

http://chemwiki.ucdavis.edu/Analytical_Chemistry/Instrumental_Analysis/Diffraction/Powder_X-ray_Diffraction.

For volume of cell calculated by:

$V = 0.866\ a^2 c$

Here,

V=volume of cell.

Density of sample relates with molecular mass, volume of cell is shown by:

$\rho = 2M/N_A V$

Here,

ρ=density of sample.

N_A= Avogadro number.

V=volume of cell.

Advantage of X-ray powder diffraction (XRD):

- Determine the unit cell volume.

- Measure the   pureness of sample.
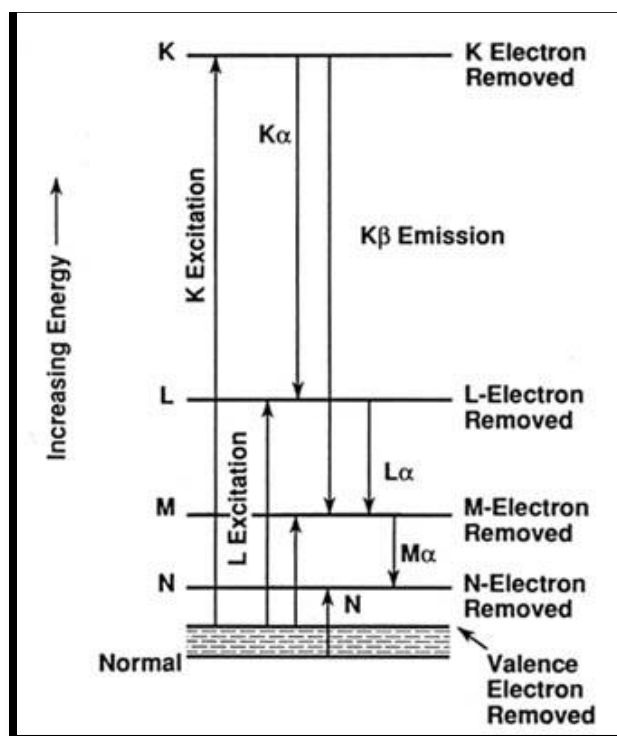
- Minimum amount of sample required.

Fig. 1: Principles of X-ray generation, reproduced from the website

Image-credit:internet-link

http://serc.carleton.edu/research_education/geochemsheets/xrays.html

2. Thermo- gravimetric analysis:

TGA study is the almost usually applied for thermal effect on the sample. It is founded that the mass (%) loss of sample as a role of high temperature. In (TGA) study, unbroken graphical record of mass (%) change w.r.t temperature is gotten after a sample is heated at a undeviating rate of temperature. Graph between, mass (%) change against temperature (T) is expressed as Thermo-gravimetric curve, better known as TG curve as shown in the Fig.2.
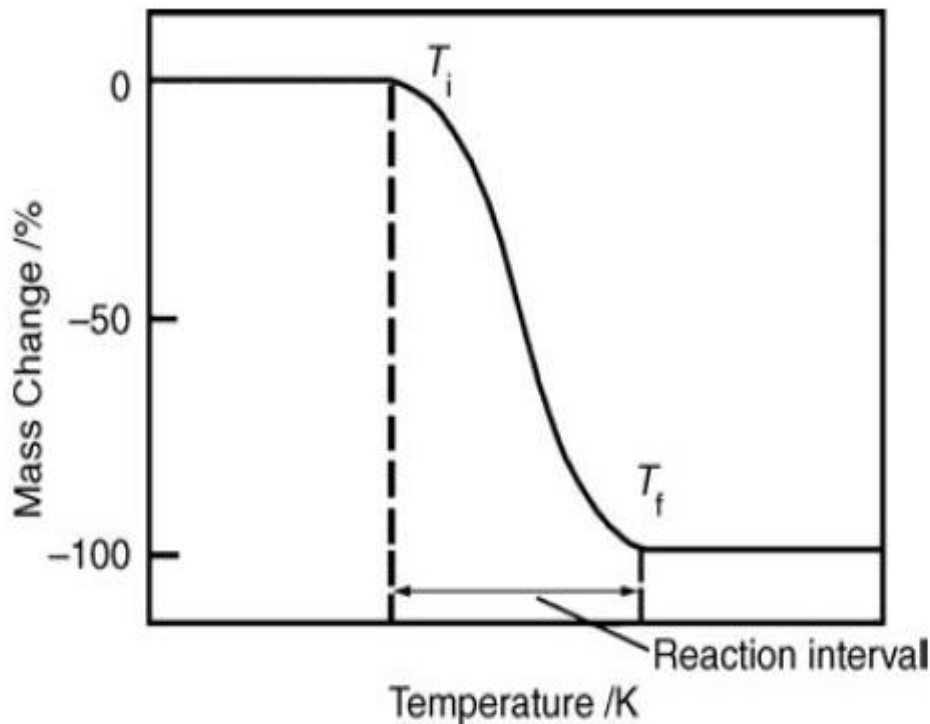
Fig 2: A simple TG curve (Leng, 2008)

The TG curve shown in above figures plotted with the mass (m) along y-axis and the temperature (T) along on the x-axis.TG curves assist in telling the level of purity of logical samples and determining the mode of their transformations within defined temperature range. In this graph during single stage decomposition of sample - there are two typical temperatures viz. the initial temperature T_i and the final temperatureT_f. $T_i$is defined as the lowest temperature at which the approaching a mass change can be found by the thermo-balance operating under particular conditions.$T_f$is defined as the final temperature at which the decomposition seems to be finished. Although, T_i has no straightforward meaning, it still has useful characteristic of the TG curve and the term procedural decomposition temperatureis indicated for T_i. The difference

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$T_i - T_f$ is termed as reaction interval. TGA study gives about physical & chemical changes that occur in sample w.r.t temperature. Physical effects are like second order phase transition, sublimation, absorption, desorption & including vaporization. Chemical phenomena are also studied such as desolation, dehydration, decomposition, chemisorption & oxidation or reduction of a given sample of reaction.

3. Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry (DSC) postulates the heating sample at controlled rate and demonstrates the heat flow either into or from the sample. DSC techniques have the same aim viz. to examine thermal effects on a sample by heating or cooling without mass exchange with its surroundings. The thermal events studied by DTA and DSC permit solid phase transformation, glass transition temperature, crystallization, observe fusion, oxidation and melting. 'Differential' stress is that analysis which is based on differences between sample material and a reference material in which the studied thermal events do not occur. The transition of phase from amorphous solid to crystalline solid is an exothermic process. If temperature of the sample is raised then sample reaches to its melting point temperature.
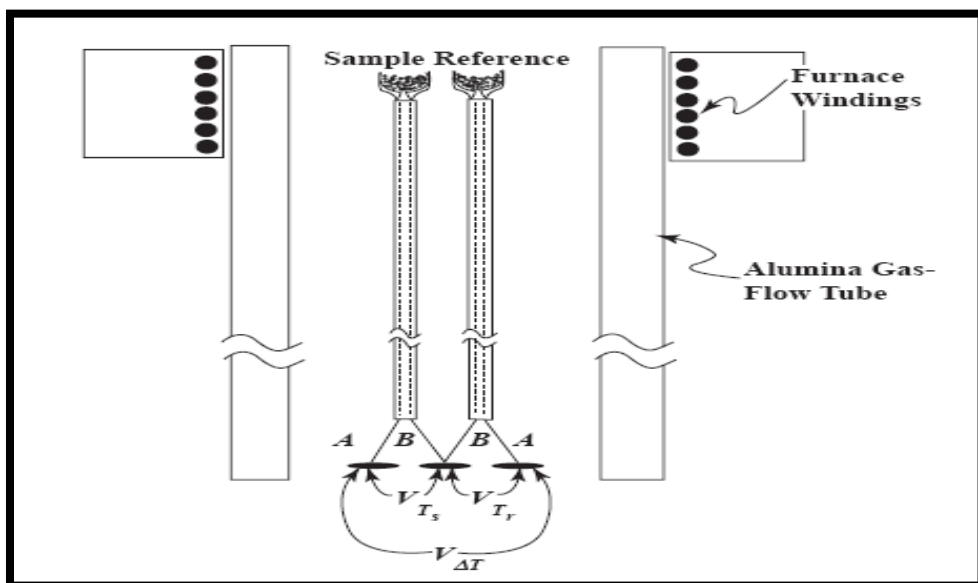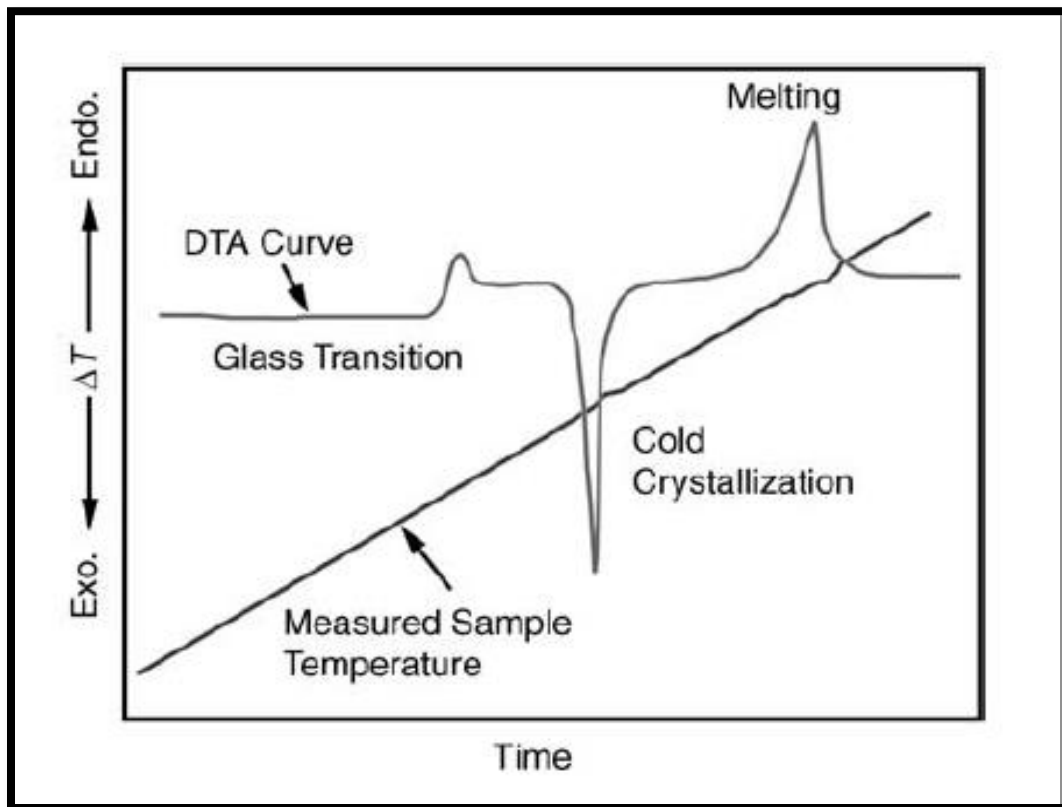

Fig 3: Schematic Diagram of DTA instrument [14]

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Fig. 4: DTA curve [14]

4. Fourier Transform Infra-Red Spectroscopy (FTIR):

This method is used for finding the functional group like in attraction between the organic-organic compounds. Fourier transform infrared spectroscopy (FTIR) is based on principal of Michelson interferometer experiments. FTIR is infrared spectroscopy in which the Fourier transform method is used to find an infrared spectrum in a whole range of wavenumbers simultaneously. It is differs from the dispersive method, which creates a spectrum by collecting signals at each wavenumber separately. Now days, FTIR is almost totally changed by the dispersive method because FTIR has a much higher signal-to-noise ratio than that of dispersive method. [14]
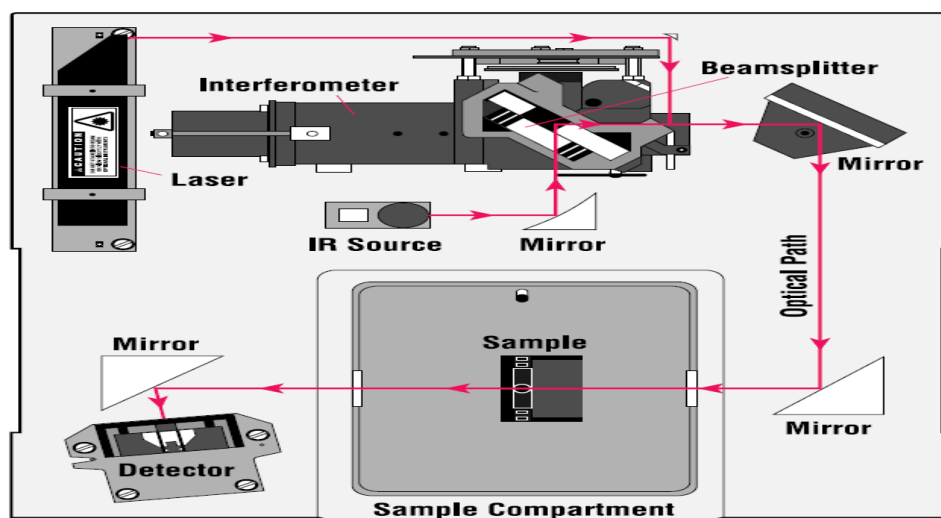
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Fig 5: Spectrometer layout, reproduced from the website

5. Vibrating Sample Magnetometer (VSM):

A vibrating sample magnetometer (VSM) works on Faraday's law of induction principle, which tells us that a varying magnetic field produces an electric field. This electric field can be calculated which says the information about the changing magnetic field. VSM study is generally for the purpose of measuring the magnetic behavior of magnetic materials, the schematics diagram of which is shown in Fig. 4.

A VSM works firstly to locate the sample to be studied in a constant magnetic field. If the sample is magnetic in nature, this applied constant magnetic field will be magnetize the sample by aligning the magnetic domains in same direction of field or the individual magnetic spins of moments with the field. The larger the value of constant field, the larger will be the magnetization. The magnetic dipole moment of the sample produces magnetic field around the sample which is known as the **magnetic stray field**. As system starts, the sample oscillates up and down; this magnetic stray field varies as a function of time and can be sensed by a set of pick-up coils.
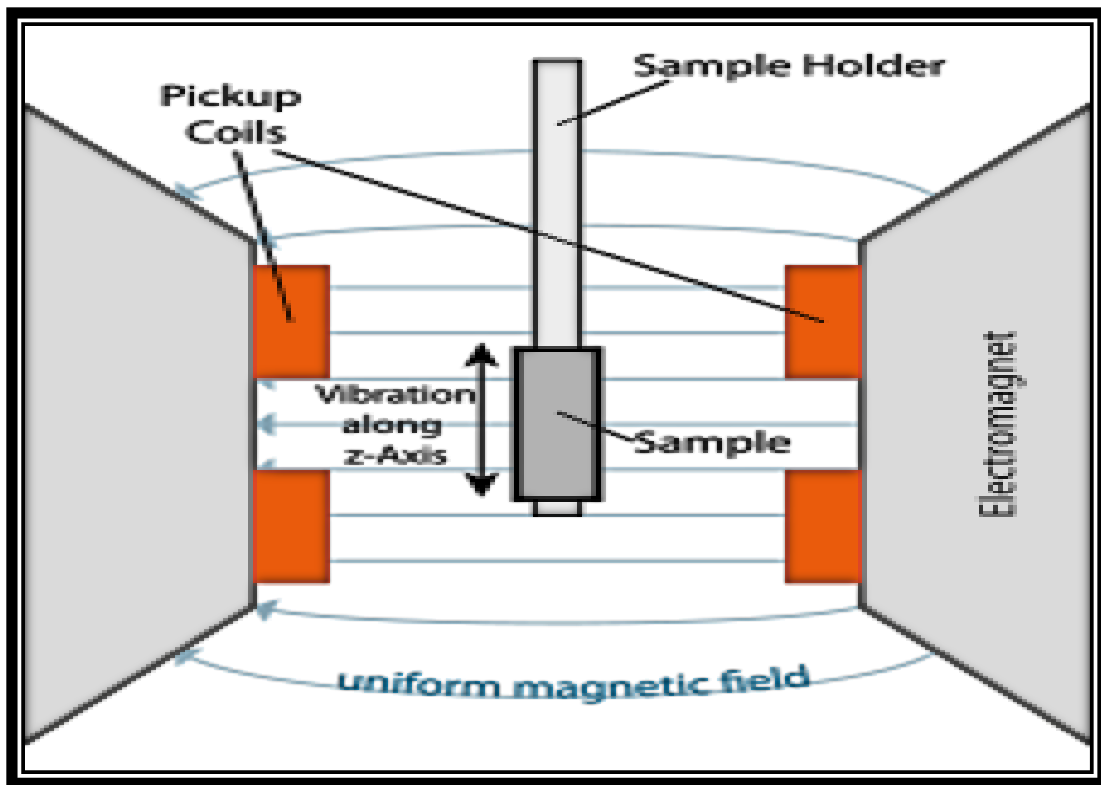
Fig 4: Schematic diagram of the Vibrating Sample Magnetometer [14]

Image-credit: internet-link:

http://en.wikipedia.org/wiki/Vibrating_sample_magnetometer.

The varying magnetic field causes an electric field in the pick-up coils according to Faraday's Law of Induction. This current is directly proportional to the magnetization of the sample. Larger the magnetization greater is the induced current. The induced current is amplified by a Trans-impedance amplifier and lock-in amplifier. The several components are fixed up to a computer interface. Controlling and monitoring software in the system can tell about how much the sample is magnetized and how its magnetization depends on the strength of the constant magnetic field

Results and discussion:

The final results of the completed project are still under observations.

1. **FTIR Results and Study:**
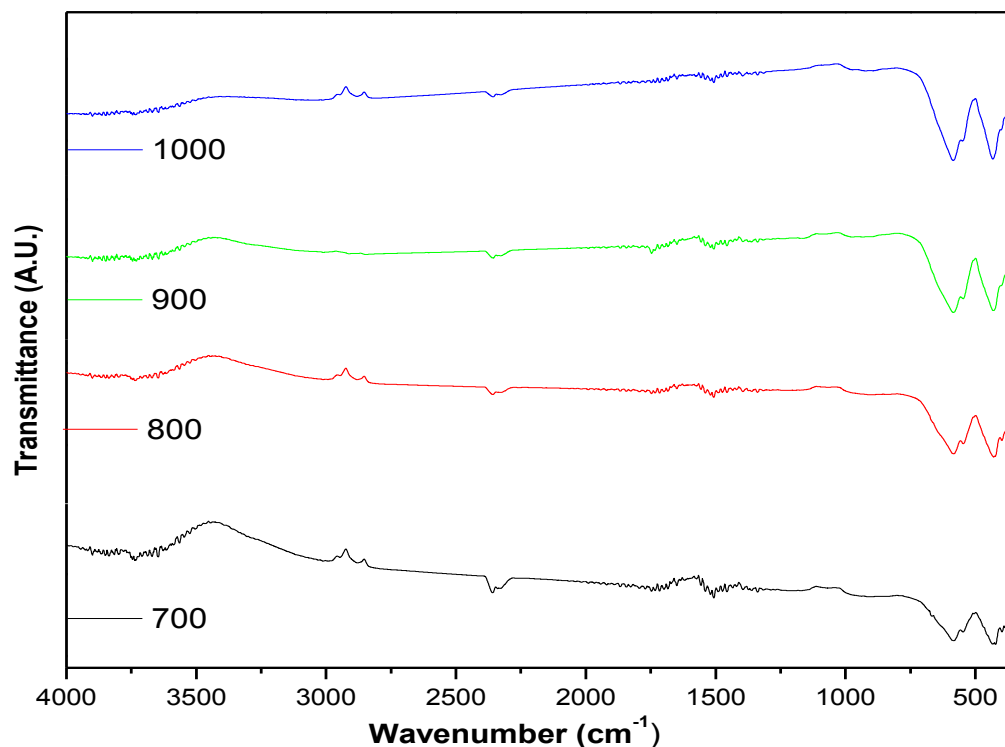
Graphs of Sample BaPrCM, FTIR, between Absorption transmittance V/s Wavenumber.

To carry out the FTIR study of the prepared powdered samples, the prerequisite is to convert the powdered samples into thin pellets using a KBr press. For this conversion of the powdered sample into pellets, a mixture of the powdered sample and KBr, in the ratio 1:10 is grinded with mortar and pestle. This powdered mixture is then pressed to form a pellet. The spectra of the samples are collected in the range of 400 to $4000 cm^{-1}$ with IR prestige-21 FTIR (model-8400S).

The bands in the range $3200\text{-}3700 cm^{-1}$ and $1600 cm^{-1}$ are assigned to the hydroxyl and carboxyl groups of the citric acid. The as-burnt powders also show some bands below $600 cm^{-1}$. This corresponds to magnetite. The band at $1380 cm^{-1}$ corresponds to the presence of NO^{-3} ion.

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This appears in samples calcined at 500°C and 600°C but vanishes in samples calcined at temperatures 700, 800 and 900 $^{\circ}$C .

The band at 1496cm^{-1} is distinguishing property of the presence of CO_3^{2-} anion. The sharp peak localized at 960 cm^{-1} is assigned to deformation vibration of C–H group. In the range 1000–100 cm^{-1}, the IR bands of solids are allocated to vibration of ions in the crystal lattice. In this case, two main broad metal-oxygen bands are visible in the IR spectra of ferrites. The highest is usually located in the range of 600–550 cm^{-1}[1] and corresponds to stretching vibrations of metal ions at the tetrahedral site, whereas the lowest, observed in the range 450–370 cm^{-1} is assigned to stretching vibrations in octahedral sites. We can conclude that after completion of the self-combustion reaction, the resulting powder's main components are metal oxides and carbonates, but a small amount of unreacted organic material is also found.

2. XRD-Results and Study:

The X-ray diffraction (XRD) patterns are taken by using Par analytical X-ray diffraction unit (X-Pert Pro) diffract meter using Cu-Kα radiation at 1.54060 Å. The XRD patterns of $Ba_{1-x}Pr_xCo_xFe_{12-x}O_{19}$ demonstrate that all the sample of the series have magneto-plumbite structure and only a small trace of $Fe_2O_3$is detected. The presence of the Fe_2O_3 is related to an incomplete calcinations in the reaction [1]. The diffraction patterns consist of peaks corresponding to crystallographic planes (006), (107), (110),(203),(205),(206),(200),(209),(220),(217) and (112) in sample having composition (x=0.4). Similarly, the peaks in diffraction patterns corresponding to the following crystallographic planes (106),(110),(114),(205),(207), (220),(112) and (217),(220) are observed in samples having variation of temperature in the range of (700-1000°C).this provides a clear evidence for hexagonal structure of doped barium hexaferrite. The observation is that the peaks of XRD pattern for the calcined powder of doped barium hexaferrite are at the same place as for the undoped barium hexaferrite but different intensities.in the doped ferrite case, the dopants of Pr^{3+},Co^{2+} are rearranged in the hexagonal structure to fulfill the formation of single hexagonal phase.

Parameters i.e. cell constant (a and c),cell volume (V),X-ray density and crystallite size (D) are calculated by using the following formula:

$1/d^2 = 4/3\ (h^2+hk+k^2)/a^2+l^2/c^2$[1]

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$V = 0.866a^2c$

$\rho = 2M/N_A V$

$D = K\lambda/\beta\cos\theta$

Where λ is the X-ray wavelength,β is the half peak width ,K=1,the constant ,M the molecular mass of the sample and N_A Avogadro's number, ρ is the density of sample,(h,k,l) are miller induces of crystal. θ is the angle of diffraction.

The values of X-ray density and crystallite size are listed in table no 1.01 and 1.02.it is observed that the average grain size is between   to   nm which is smaller than that reported [2] earlier i.e. 14-30nm for the M-type hexaferrite. The density of X-ray increases with increase in concentration of the doped ion, which is lower in molecular weight of doped samples as compared to $BaFe_{12}O_{19}$, as the value X-ray density depends on the molecular weight of the sample [2].the lattice constant a and c are calculated for the prominent peaks (006) and (110) for samples with composition and (107) and (110) are calculated for the samples with temperature variation.it is found that the lattice constant a does not change drastically with the mole ratio, a slight decrease in the lattice parameter c are found. This may be attributed to the small ionic radius of Pr compared to Fe.
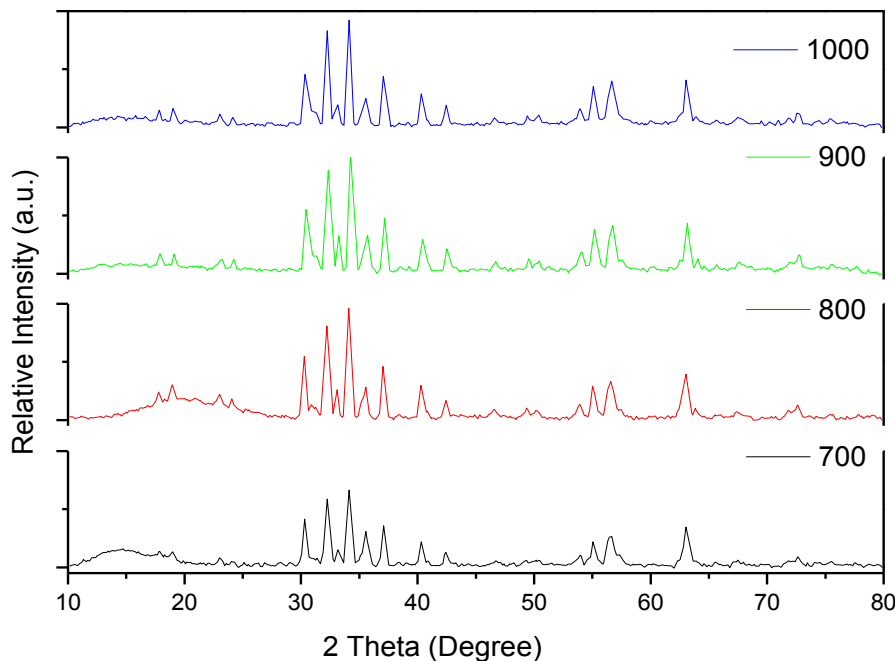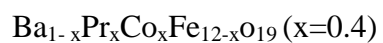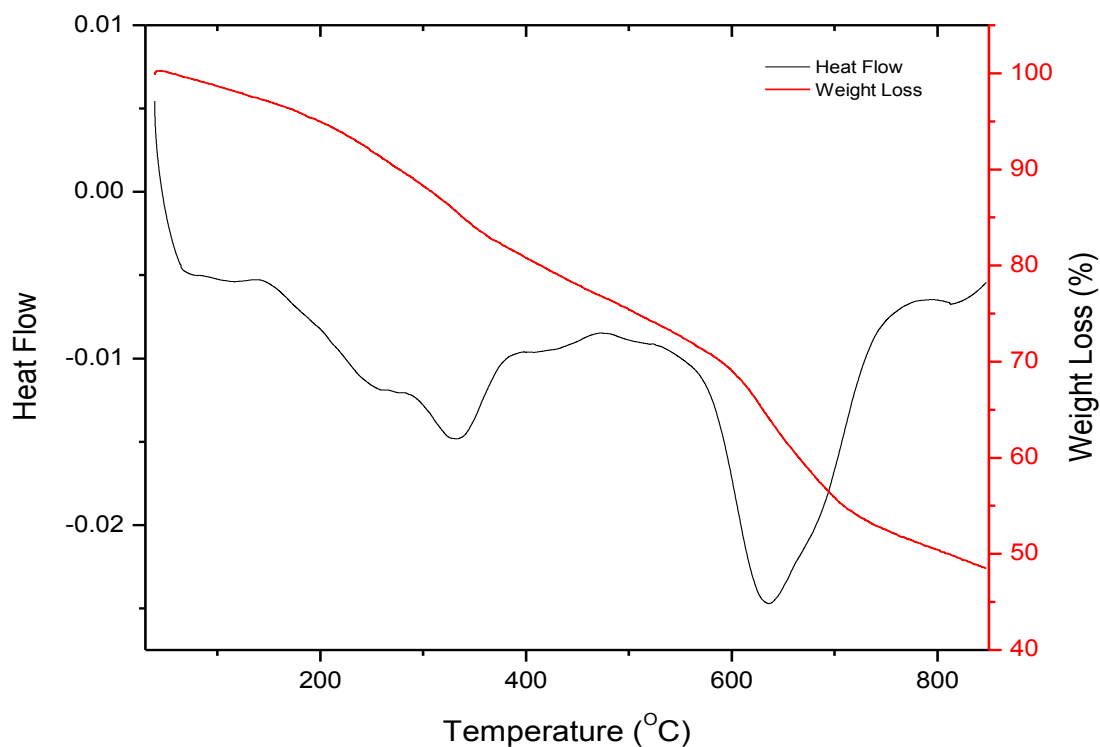

Fig .: XRD graph of sample BaPrCM , between 2θ V/s relative intensity (a.u).

Table 1.01: Average particle size, lattice constants, volume and density of

$Ba_{1-x}Pr_xCo_xFe_{12-x}O_{19}$ (x=0.4)

Sample number	Variation T=temperature (°C)	Average grain size, D (nm)	Lattice constants		$V_{CELL}=$ 0.866a^{2}c (Å^3)	X-ray density (g/cm^3)
			a(Å)	c(Å)		
1	700	33.04	4.80	23.17	462.94	2.41
2	800	36.17	4.81	23.17	464.60	2.40
3	900	43.29	4.79	23.07	459.20	2.43
4	1000	34.49	4.81	23.12	463.51	2.40

3. DSC/TGA Results and Study:

Graph of sample BaPrCM, between Heat Flow, Weight Loss (%) V/s Temperature variation.

The differential scanning calorimetric (DSC) and thermo gravimetric (TG) graphs are created for the prepared sample $BaFe_{12}O_{19}$ for analyses up to temperature 820^0C in an argon gas to observe the structural changes such as loss of weight of sample and shift different phases during heat treatment. The two peaks of endothermic are appeared in the DSC curve at a temperature about 469.7 and 783.03^0C. The single M-type hexaferrite is not formed directly by sol- gel auto combustion method. The gasses like CO_2 and CO produced during auto combustion process, are due to decomposition of citric acid .The $(BaCO_3)$Barium carbonate formed during the combustion process are due to interaction between very reactive Ba^{2+} ions and CO_2 or CO. These first endothermic peak at 469.7^0C credit the formation of metal oxide from hydroxide to the decarboxylation of $BaCO_3$[1, 2].The second endothermic peaks shows the formation of mono ferrite phase. [3]This is at temperature range of 331.6 and 635.8°C respectively. First exothermic peak gives the drastic mass loss, due to autocatalytic oxidation reduction reaction between metal nitrates and citric acid. The TG curve shows two steps of weight loss in % .finally the formation of M-type hexaferrite phase from the monoferrites and metal oxides. This shows that the prepared hexaferrite developed at this temperature. The obtained the M-type hexagonal phase as also confirmed by the XRD analysis.

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Reaction interval= (T_F-T_i)= (724.2-184.4)°C=539.8°C.

4. VSM Results and Study:

Vibrating Sample Magnetometer is used to study the magnetic properties of the Nano –hexaferrite .At room temperature, the hysteresis loops of the different ferrite sample were analyzed by applying magnetic field 15kOe using VSM (Model PAR 155, from Princeton Applied Research USA).This study is carried out in order to take the measurement of the different magnetic parameters, like saturation magnetization (Ms), retentively and coercivity (Hc), anisotropy of the Nano-hexaferrite as shown fig: 1.

For calculating anisotropy of sample we use formula:

$$H_c = \frac{2K}{\mu_o M_s} \quad . \quad [1]$$

Here Hc is coercivity is anisotropy, MS is saturation magnetization & μ_0=4π x10^{-7}H/m is permeability of free space. It is observed that with increase in temperature, the value of saturation magnetization reduces and also the value of the retentively, coercivity and anisotropy. The increase in the coercivity is due to fact that the grain size increase from super-magnetic to single domain range. The value of saturation magnetization, retentively, coercivity and anisotropy are shown in table 2.1.
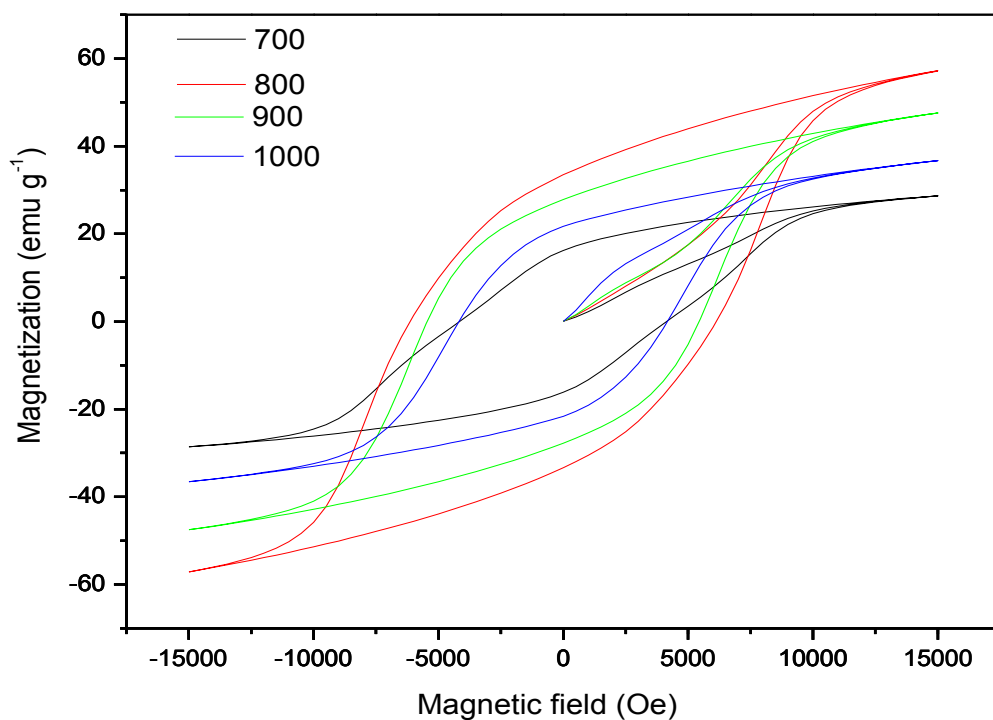
Graph of sample (BaPrCM) at different range of temperature between magnetization (emu/g) V/s Magnetic Field (Oe).

Table 2.1:

The hysteresis loop of concentration variation (x=0.4) $Ba_{1-x}Pr_xCo_xFe_{12-x}O_{19}$.at different temperature.

S.No.	Temperature Variation (°C)	Saturation Magnetization (Ms) (emu/g)	Retentively (emu/g)	Coercivity(Hc) (Oe)	Anisotropy (K)
1	700	28.64	16.17	4000.38	71.9×10^{-3}
2	800	57.15	33.40	6500.39	233.3×10^{-3}
3	900	47.60	27.78	5500.36	164.4×10^{-3}
4	1000	36.69	21.66	4500.39	103.7×10^{-3}

Conclusion and Future Scope:

The formation of sample BaPrCM with doped rare earth elements properly and their no exist any impurity exist in sample which are explained by XRD,TGA/DSC analysis. XRD study proves its hexagonal Magnetoplumbite structure of single phase state transition exist.as temperature increase grain size increase first upto 900°Cafter that decrease. The DSC/TGA study says its physical and chemical transition with respect to temperature variation shows in form of exothermic or endothermic peaks, which shows heat absorbed or evolved respectively in the surrounding space. FTIR study shows the sample BaPrCM bound formation properly between dopant materials Par sodium. the VSM Study, shows its magnetic property like coercivity, magnetization and anisotropy. The sample shows greater value of coercivity and saturation magnetization at 800°C 6500.39 Oe and 57.15emu/g respectively. Therefore it is used for much application in permanent magnetic materials field of research, for many electronics media, transformer core ,Wi-Fi system due to maximum absorption of em waves materials etc.

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