

“SYNTHESIS AND MAGNETIC PROPERTIES OF COBALT FERRITE WITH DIFFERENT MORPHOLOGY”

A thesis submitted

By

Rahul Kumar Mallik

108CR042

In the partial fulfilment of the requirements of degree

Of

Bachelor of Technology



DEPARTMENT OF CERAMIC ENGINEERING

NATIONAL INSTITUTE OF TECHNOLOGY

ROURKELA

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CERTIFICATE

This is to certify that the thesis entitled, “Synthesis and magnetic properties of cobalt ferrite with different morphology”, submitted by **Mr. Rahul Kumar Mallik (Roll no. 108CR042)** in partial fulfillment of the requirements of the award of Bachelor of Technology Degree in Ceramic Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other university / institute for the award of any Degree or Diploma.

Date: 11.05.2012

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Rahul Kumar Mallik

108CR044

Table of Contents

Chapter 1: Introduction	1
1.1 Introduction	2
Chapter 2: Literature Review.....	4
2.1 Review.....	5
2.2Different synthesis route	6
Chapter 3: Experimental Procedure.....	8
Chapter 4: Result & Discussion.....	16
Chapter 5: conclusions	30
Chapter 6: References	32

ABSTRACT

The cobalt ferrite was synthesized by wet precipitation method using cobalt chloride and Iron chloride solution with Hydrazine Hydrate precipitating agent. The precipitation was also carried out in the presence of surfactant CTAB to observe the change in magnetic properties and morphology of the samples. Different solvent were also used like di-ionized water and Ethylene Glycol for the precipitation process. The above obtained powder was calcined at different temperature (600°C and 800°C) and also sintered at different temperature (1000°C and 1200°C). Different characterization like SEM was done to visualize the morphology and particle size. The XRD analysis was done to know the phases present and the B-H curve to get the magnetization and corecivity of the powder. The corecivity decreased with the increase in sintering temperature and varied from 200-900 Oe. The XRD acquired showed pure phase of cobalt ferrite.

List of tables and Figures:

Figure 1 :- SEM IMAGE OF HIS	17
Figure 2 :- SEM IMAGE OF HIS	17
Figure 3:- XRD OF SAMPLE HIS	18
Figure 4 :- B-H curve of sample HIS	18
Figure 5:- SEM image of sample SIH.....	19
Figure 6:- SEM image of sample SIH (higher magnification).....	19
Figure 7:- XRD of sample SIH calcined at 600°C.....	20
Figure 8:- XRD of sample SIH sintered at 1000°C.....	20
Figure 9:- B-H curve of sample SIH calcined at 800°C and sintered at 1000°C.....	21
Figure 10:- B-H curve of the sample SIH calcined at 800°C and sintered at 1200°C.....	21
Figure 11:- XRD of sample C3 calcined at 800°C.....	22
Figure 12:- B-H curve of sample C1 calcined at 800°C and sintered at 1000°C.....	22
Figure 13:- B-H curve of sample C1 calcined at 800°C.....	23
Figure 14:- B-H curve of sample C3 calcined at 800°C and sintered at 1000°C.....	23
Figure 15:- B-H curve of the sample C3 calcined at 800°C.....	24
Figure 16:- SEM image of sample HISCO	25
Figure 17:- SEM image of sample HISCO	25
Figure 18:- B-H curve of the sample HISCO calcined at 800°C.....	26
Figure 19:- B-H curve of the sample HISCO calcined at 800°C and sintered at 1200°C.....	26
Figure 20: SEM image of sample HISG powder (low magnification)	27
Figure 21: SEM image of sample HISG powder (high magnification)	27
Figure 22: SEM image of sample HISG powder (high magnification)	28
Figure 23: B-H curve of HISG calcined at 800°C	28

CHAPTER 1

I**NTRODUCTION**

1.1 Introduction

Ferrites are chemical compounds which are composed of a ceramic material and Iron oxide as their main component. A ferrimagnetic ceramic compound, ferrites, has a spinel type structure. The magnetic property of the ferrite is due the structure and the distribution arrangement of the ions in the sub lattice. Most of the ferrite have a spinel structure with a formulae AB_2O_4 , where “A” are divalent ions such as Mg^{2+} , Co^{2+} , Ni^{2+} , Mn^{2+} , and “B” are the trivalent ions such as Fe^{3+} and Al^{3+} . Spinel structure have an oxygen ion sub lattice, in a cubic close-packed arrangement with cations occupying various combinations of the octahedral (O) and tetrahedral (T) sites. The cubic unit cell contains 8 formula units and containing 32 O and 64 T sites. Spinel are basically categorised into a normal and inverse spinel. In normal spinel, the divalent cations “A” are positioned at the tetrahedral (T) sites and the trivalent cations “B” on the octahedral (O) sites. $NiFe_2O_4$ (NF) or $CoFe_2O_4$ (CF) has an inverse spinel crystal structure. In inverse spinel “A” cation occupies one half of the octahedral coordination sites and half the “B” cation occupies the other half (O) sites as well as all “T” sites. In spinel structure the atom A and atom B are exactly antiferromagnetic, the spin cancel each other. The magnetic property rise due to the $4 Fe^{+2}$, which align themselves on the application of field. Depending on the magnetic properties, ferrites can be categorized as “soft” and “hard” ferrite. Soft ferrites have low corecivity while the hard ferrites have high corecivity. Hard ferrite have high corecivity and moderate magnetization .corecivity stands for the resistance to get demagnetized on the removal of the applied field which satisfy it for being a permanent magnet ^[1].

Cobalt ferrite:

Cobalt ferrite is categorised into a hard magnet due to its high corecivity and moderate magnetization. Due to its high magnetic corecivity value and good physical and chemical stability it has been used for various applications. Cobalt ferrite ($CoFe_2O_4$) neither has a

spinel or invers spinel structure. It has partially inverse spinel structure $[\text{Co}_k^{2+}\text{Fe}_{1-k}^{3+}](\text{Co}_{1-k}^{2+}\text{Fe}_{1-k}^{3+})\text{O}_4$, having a corecivity value of 1000 Oe and moderate magnetization of 50 emu/g. Due to its high value, they become a perfect for using in high density magnetic storage materials, ferrofluids, medical diagnosis, magneto-mechanical, and torque sensors.

CHAPTER 2

LITERATURE

REVIEW

2.1 Review

Magnetic nanoparticles have been the topic of strong research because of their possible applications in high-density magnetic recording, magnetic fluids etc.^[2-4] Amongst the several ferrite materials for magnetic recording applications, cobalt ferrite (CoFe_2O_4) has been widely considered because it retains excellent chemical stability and good mechanical hardness^[5]. In addition to the detailed control on the composition and structure of CoFe_2O_4 , the achievement of its practical application depends on the ability of controlling crystal size within the super paramagnetic and single domain limits. It is identified that the crystal size is related to the relative interdependence among the nucleation and growth steps, which in turn can intensely be affected by the solution chemistry and precipitation conditions. Depending on the above basis, the research contains a modified coprecipitation synthesis method where the growth of cobalt ferrite crystals is regulated to achieve high room-temperature coercivity and moderate magnetization at the nanoscale. This size-controlled synthesis method became possible by adjusting the oversaturation conditions and reaction time in the course of ferrite formation in aqueous solutions^[6-8].

The necessity for advanced automated systems in engineering is increasing due to the necessity to scale up production. Most automated systems depend seriously on high sensitivity sensor or energy efficient actuator devices. Stress sensors and actuators can be advanced by exploiting the magnetomechanical coupling capability in magnetostrictive materials. It is significant to improve the properties of these materials for such uses. This has sparked significant research interest in magnetostrictive materials in developing devices for unlike applications, especially where non-contact process is critical. Non-contact process capability is very useful for devices because accurateness, repeatability and linearity are tainted over time due to wear and tear in devices functioning in contact mode. Appropriate magnetostrictive materials for such devices should have sufficient amplitude of

magnetostriction for the proposed applications and high sensitivity of magnetostriction to functional magnetic field (strain derivative). Materials built on cobalt ferrite are candidates for such device progress because of their wanted magnetostrictive properties. Furthermore, since it has been shown that magnetostriction amplitude of as low as 30 ppm is enough for numerous uses, the typical amplitudes of magnetostriction, 100–225 ppm, stated for cobalt ferrite in numerous studies are highly sufficient for many applications^[9-12].

As a outcome, sensor and actuator designs built on cobalt ferrite are likely to be durable, chemically stable and show good response to stimulus in use. In order to attain materials with desired properties, it is required to obtain high-density powders with small and uniform grain size, and precise stoichiometry. This goal is attained more easily using wet chemical methods such as coprecipitation, spray drying, hydrothermal synthesis and sol–gel process. It is recognized that a powder is made up of an aggregation of primary particles, which modify their characteristics according to the process of preparation and/or type of drying. A powder as set can cultivate inherent porosity after drying and then remove these pores after thermal treatment with a rise in its density. Cobalt ferrite is an essential material not only for its magnetic properties but also for its catalytic properties which depends on the textural and morphological characteristics. This kind of ferrite is a spinel but it shows a large corecivity inversely from the rest of the spinel ferrites. In the present investigation, nanocrystalline CoFe_2O_4 powders have been arranged using metal nitrates in aqueous media and adding ammonium hydroxide to destabilize the solution. It has focused the attention on the morphological changes of the powders with calcination temperature^[13-16].

2.2 Synthesis of cobalt ferrite through different route

Ceden o-Mattei et al [2] has worked on synthesis of high corecivity of cobalt ferrite by co precipitation method. In this work, cobalt ferrite was synthesised by proper control on the circumstances during the ferrite formation in aqueous phase. The effect on crystal growth was

resulted in improvement of coercivity value from 237 Oe, with no control of flow-rate, up to 1337 Oe, with controlled flow rate.

Juliana et. al. [13] has worked to observe the behaviour of heat treatment on nano particle cobalt ferrite powder. In his work, using Fe and Co nitrates as precursors, and ammonium as a precipitant of manufacturing, of cobalt ferrite monophasic, ultrafine and pure particles was successfully done. With suitable heat treatment it is possible to control the textural and magnetic characteristics of cobalt ferrite as desired. It showed that parameters such as density, BET C constant, total pore volume were dependant on the morphology and magnetic property.

CHAPTER 3

EXPERIMENTAL

PROCEDURE

Experimental procedure 1:

2.379 gram of cobalt chloride was taken in a 100 ml beaker and was added with 20 ml of deionised water to prepare a solution of cobalt chloride with .5 molar concentrations. It was taken separately in a beaker and was stirred in a magnetic stirrer (for 1 hour) to get a clear solution.

In another 100 ml beaker was taken 3.3 grams of iron chloride with 20 ml of de-ionized water to prepare a solution of iron chloride of 1 molar concentration and was stirred in a magnetic stirrer (for 1 hour) to obtain a clear solution. Then both the solutions were transferred to a single beaker and stirred (for 1 hour) to get a homogeneous solution. 2 molar concentration of ascorbic acid (7.04 grams in 20 ml de-ionized water) was taken in a 50 ml burette and was added to the solution prepared, drop wise from the top with help of a burette stand while stirring. The colour of the solution changed from light red to dark red after stirring the solution for 2 hours without heating. The pH of the solution was observed to be between 2 and 3.

Experimental procedure 2:

2.4 gram of cobalt chloride was taken in a 50 ml beaker to which 20 ml of de-ionized water was added and stirred for 1 hour to get a clear red solution of cobalt chloride. In another beaker 3.3 gram of iron chloride was added in 20 ml de-ionized water in a 50 ml beaker for 1 hour to get a green colour clear solution. Both the solution were then transferred to a 100 ml beaker and stirred for 1 hour to make the mix homogeneous. Ascorbic acid was added from the top with help of a burette and was stirred slowly with help of the magnetic stirrer. The pH of the solution was increased by adding NH_4 drop wise to reach a pH level of 12- 13. No observation of precipitation was found. The colour of the solution changed from dark red to black. The solution was left for ageing (24 hours). It was then heated at 70°C with help of

the magnetic stirrer for 1 hour. A black colour coating was found on the solution which was removed and dried. The solution was filtered with help of a filter paper (40) and a dark hard precipitate was obtained. The XRD analysis of the precipitate was also carried out from 20°-60° for 10 minutes.

Experimental procedure 3:

Precursors used:

- 1) .5 molar cobalt chloride (20 ml)
- 2) 1 molar iron chloride (20ml)
- 3) Hydrazine (20ml)
- 4) De-ionized water

Apparatus required:

- 1) 2 beaker -50 ml
- 2) 1 beaker -50 ml
- 3) Magnetic stirrer
- 4) Burette
- 5) Filter paper
- 6) Funnel
- 7) Litmus paper

Procedure:

2.4 grams of cobalt chloride was added in 20 ml de-ionized water and was taken in a 50 ml beaker and stirred (1/2 hour) to produce a reddish colour clear solution of .5 molar concentration of cobalt chloride. 1 molar concentration of iron chloride was prepared by taking 3.4 grams of iron chloride in 20 ml de-ionized water and was stirred in a 50 ml beaker

(1/2 hour). Then both the solution was transferred to a single beaker (100 ml) and was stirred in the magnetic stirrer (1 hour). With the help of burette (from top) hydrazine hydrate was added to the solution drop wise with continuous stirring. After addition of the Hydrazine Hydrate to the salt solution the solution was stirred with the help of a magnetic stirrer (for 2 hours) and the pH of the solution was found to be 11. A brown precipitate was observed at the bottom of the beaker. The precipitate was washed with hot water (4 times) to get a pH 7. Then the precipitate was filtered through a litmus paper (40) slowly. The filtered product was dried under the IR lamp to get a brown coloured powder on the litmus paper which was scratched out from it. The obtained powder was calcined at 600° C/ 2 hours. The XRD of the calcined powder was done to find out the different phases present.

SAMPLE NAME:- HIS

Experimental procedure 4:

0.5 molar concentration cobalt chloride solution was prepared by adding 2.4 grams of cobalt chloride in 20 ml of de-ionized water in a 50 ml beaker and stirring (1/2 hour) in the magnetic stirrer.

1 molar concentration of iron chloride solution was prepared by adding 3.4 grams of iron chloride in 20 ml de-ionized water in a 50 ml beaker and stirring (1/2 hour) it in a magnetic stirrer.

Then both the solutions were transferred into a 100 ml beaker and stirred together for 2 hour with the help of a magnetic stirrer to get a homogenous solution. The colour of the solution that was obtained was dark red colour.

The above solution obtained was taken in a burette (50 ml). Hydrazine hydrate was taken in a 100 ml beaker (20 ml) and the salt solution was added to it from the top with continuous

stirring. After addition of the 40 ml salt solution in the hydrazine a brown colour solution was obtained which was stirred in the magnetic stirrer (2 hours). The above solution obtained was left for precipitation for 1 night and a brown colour precipitate was obtained. The precipitate was then washed with hot water till pH-7. Then it was filtered with help of litmus paper-40 and was dried under the IR lamp and the dried powder was scratched off the litmus paper and was collected. The powder obtained was calcined at 600°C/2hour. Then the XRD analysis of the powder was done to obtain different phases present.

SAMPLE NAME:- SIH

Experimental procedure 5:

Combustion synthesis

Precursor used

- 1) 1 molar concentration of Ferric Nitrate ($\text{Fe}_3(\text{NO}_3)_2$)- 100ml
- 2) .5 molar concentration of Cobalt Nitrate $\text{Co}(\text{NO}_3)_2$ -50 ml
- 3) Citric acid (fuel)- 31.5 grams

Procedure 1

100 ml of $\text{Fe}_3(\text{NO}_3)_2$ + 50 ml of $\text{Co}(\text{NO}_3)_2$ was taken in a 500 ml beaker and stirred for 1 hour. Then Citric acid (fuel-31.5 grams) was added to this solution which was stirred for ½ hour. pH of the solution was observed with the help of the litmus paper and was found to be 1. Heating of the solution was done at 150°C in the magnetic stirrer. After heating the solution for some time a gel was formed which turned to a xerogel, then after sometime the auto combustion occurred.

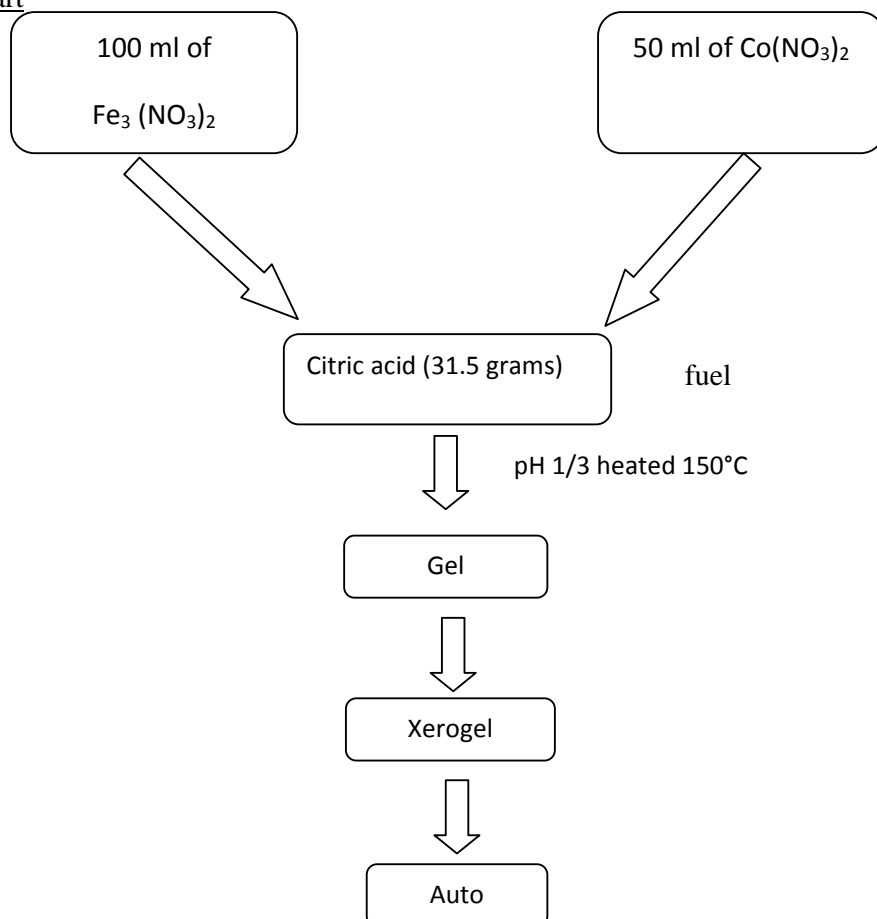
SAMPLE NAME:- C1

Procedure 2

100 ml of $\text{Fe}_3(\text{NO}_3)_2$ + 50 ml of $\text{Co}(\text{NO}_3)_2$ was taken in a 500 ml beaker and was stirred for 1 hour. Then citric acid (fuel-31.5 grams) was added to the above solution which was stirred for $\frac{1}{2}$ hour. pH of the solution was changed for 1 to 3 by adding NH_4 slowly to the above solution. Heating of the solution was done at 150°C in the magnetic stirrer. After heating the solution for some time a gel was formed which turned to a xerogel, then after sometime the auto combustion occurred. The above powder obtained was calcined at 800°C /2 hours in the furnace. The XRD analysis of the above powder was obtained.

SAMPLE NAME:- C3

Flowchart



Experimental procedure 6:

In presence of a surfactant

0.5 molar concentration of cobalt chloride was prepared by adding 2.4 grams of cobalt chloride in 20 ml de-ionized water and was stirred for ½ hour. After stirring for ½ hour surfactant (2 wt %=0.0951 gram) CTAB was added to the solution and was stirred for 1 hour. Foaming was observed in the solution.

1 molar concentration of ferric chloride was prepared by adding 3.4 grams of ferric chloride in de-ionized water (20 ml) and was stirred for ½ hour. After stirring for ½ hour, surfactant (2 wt%= 0.1297 grams) CTAB was added to the salt solution and was stirred for 1 hour. Foaming was observed in the solution.

Both the solution was transferred to a single beaker and was stirred for 1 hour in a 100 ml beaker. 20 ml Hydrazine Hydrate was taken in a 50 ml burette and was added to the salt solution from the top. A brown colour precipitate was seen after addition of hydrazine hydrate to the salt solution. The precipitate was washed once with hot water and was transferred to a petri dish, then was directly dried under the IR lamp to obtain the brown colour powder. The obtained powder was calcined at 800°C/2hour in the pit furnace.

SAMPLE NAME: - HISCO

Experimental procedure 7:

In Ethylene Glycol as solvent and surfactant CTAB

2.4 grams of cobalt chloride was added in ethylene glycol (20 ml) to prepare a solution of .5 molar concentrations. The cobalt chloride solution was stirred for ½ hour to get a clear solution in which the surfactant CTAB (2 wt%=.0951 grams) was added while stirring.

Foaming was observed during stirring. In an another beaker 3.4 grams of ferric chloride was taken with 20 ml of ethylene glycol and continuously stirred for $\frac{1}{2}$ hour after addition of surfactant CTAB. Foaming was observed while stirring. Both the solution was then transferred to a single beaker and stirred (1 hour) with the help of a magnetic stirrer to get a homogeneous solution. Hydrazine hydrate (20 ml) was taken in a 50 ml buret and was added from the top to the salt solution. A brown colour solution was obtained which was dried directly under the IR lamp to get the powder. The powder was collected and calcined at $800^{\circ}\text{C}/2$ hour. Then the B-H curve of the pellet was obtained.

SAMPLE NAME: - HISG

CHAPTER 4

RESULTS

&

DISCUSSION

PROCEDURE 3:

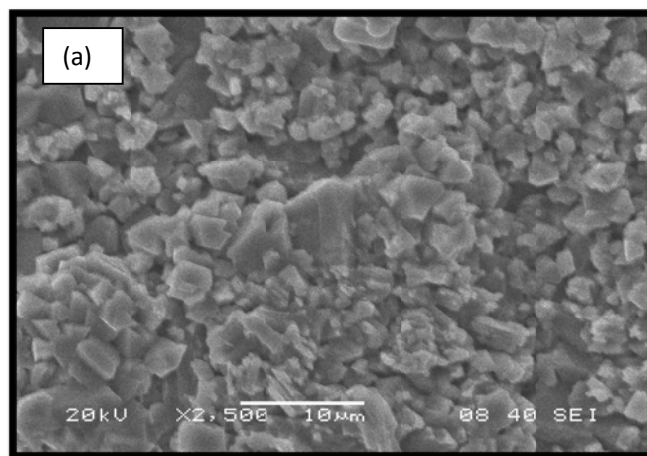


Figure 1 :- SEM IMAGE OF HIS

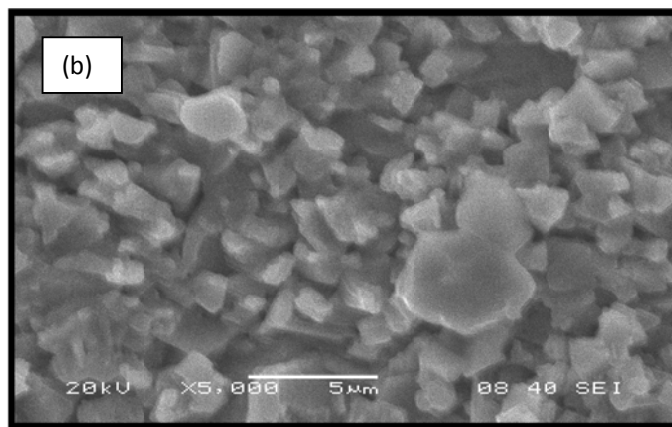


Figure 2 :- SEM IMAGE OF HIS

Fig1 & Fig 2 shows the SEM of the sample prepared in procedure 3 at different magnification. The SEM was in a SEI mode. From the SEM it can be seen that the grains have a irregular distribution and irregular shape with a average particle size of 2 μm . Particles are nearly spherical with agglomerated in nature.

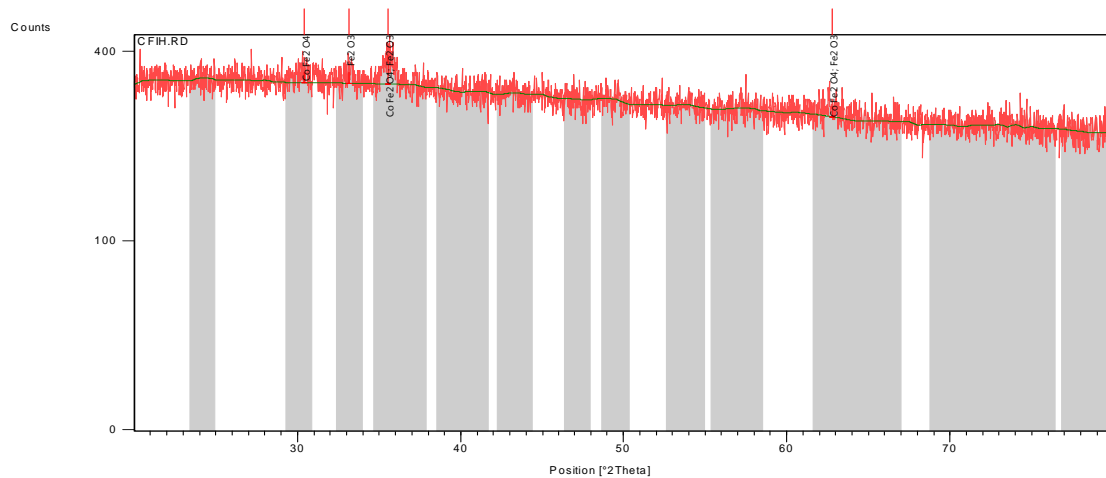


Figure 3:- XRD OF SAMPLE HIS

From the Fig. 3, the phases of cobalt ferrite (CoFe₂O₄) were obtained. Some other phases of Fe₂O₃ were also seen. The 1st peak with hkl value (220) and the 3rd peak (311) are the cobalt ferrite peak while the second peak with hkl value (104) is the ferrite peak.

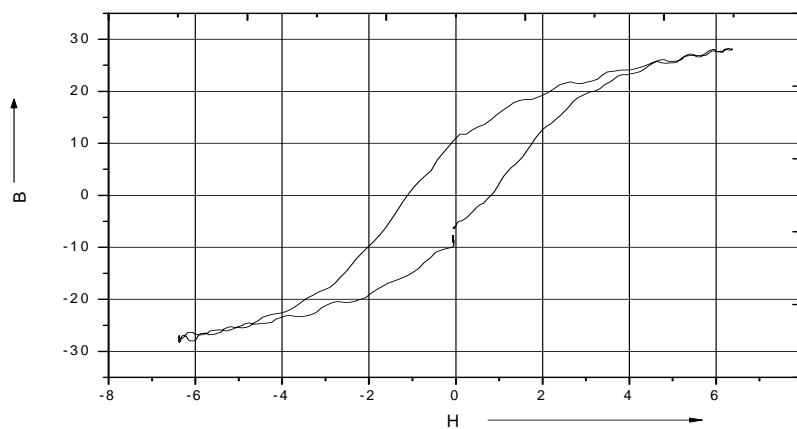


Figure 4 :- B-H curve of sample HIS

FIG 4 shows the B-H curve of the sample HIS (calcined at 600°C) which have a magnetic saturation value (M_s) about 29 emu/gram and the magnetic corecivity value about 357 Oe.

PROCEDURE 4:

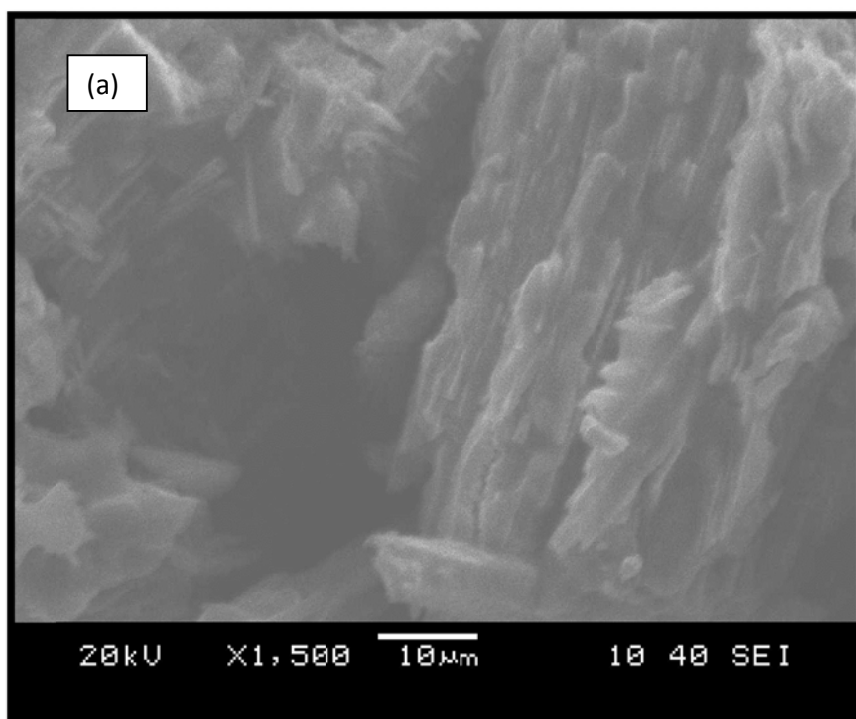


Figure 5:- SEM image of sample SIH

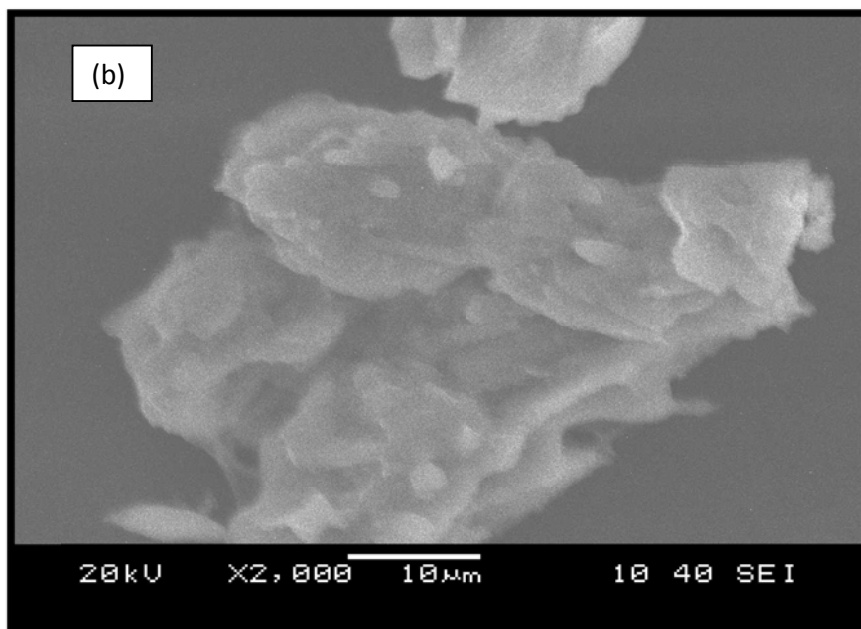


Figure 6:- SEM image of sample SIH (higher magnification)

Fig 5 and Fig 6 are the SEM image of the SIH powder sample. It can be seen that the powder are in agglomerate form. The powder samples seems to be flake in nature.

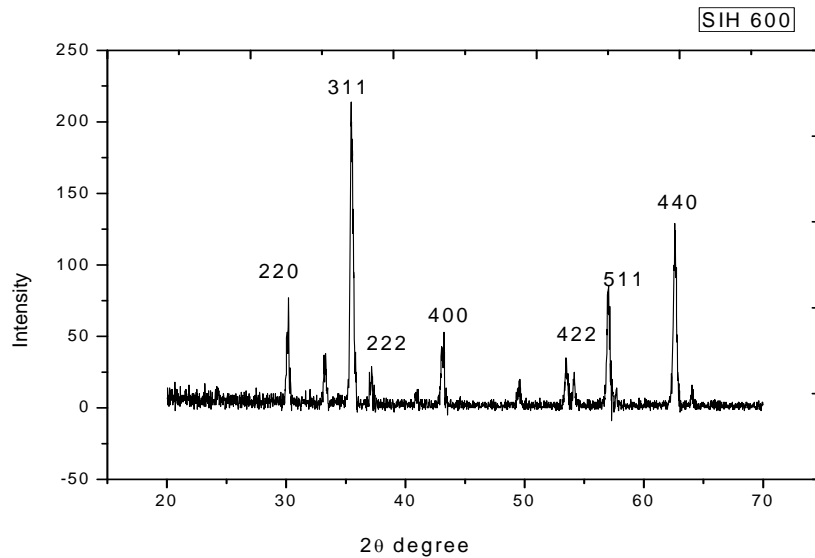


Figure 7:- xrd of sample SIH calcined at 600°C

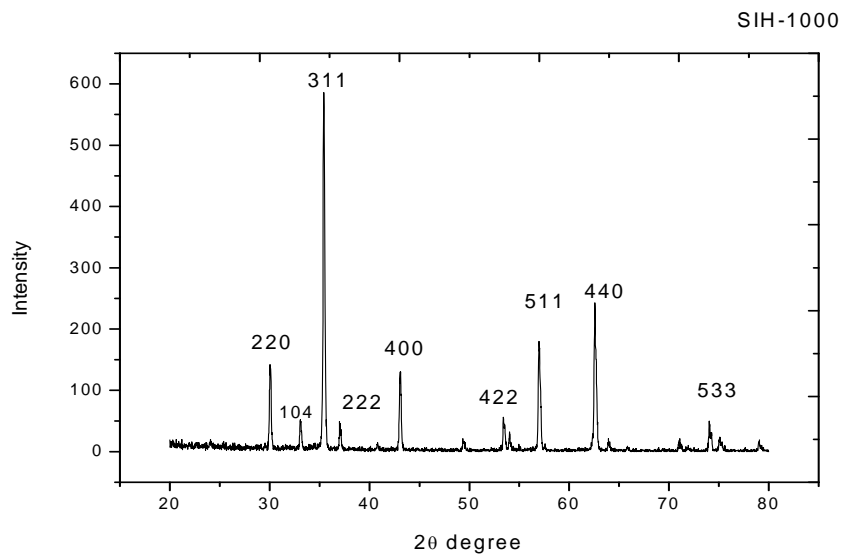


Figure 8:- XRD of sample SIH sintered at 1000°C

Fig 7 and fig 8 shows the XRD analysis of the sample SIH having phases of cobalt ferrite some phases of ferrite present in it. The peaks with hkl value, 1st peak (220), 3rd peak (311), 4th peak (222), 5th peak (400) 6th peak (422) and 7th peak (511) show the cobalt ferrite phase while the 2nd peak (104) has the ferrite phase.

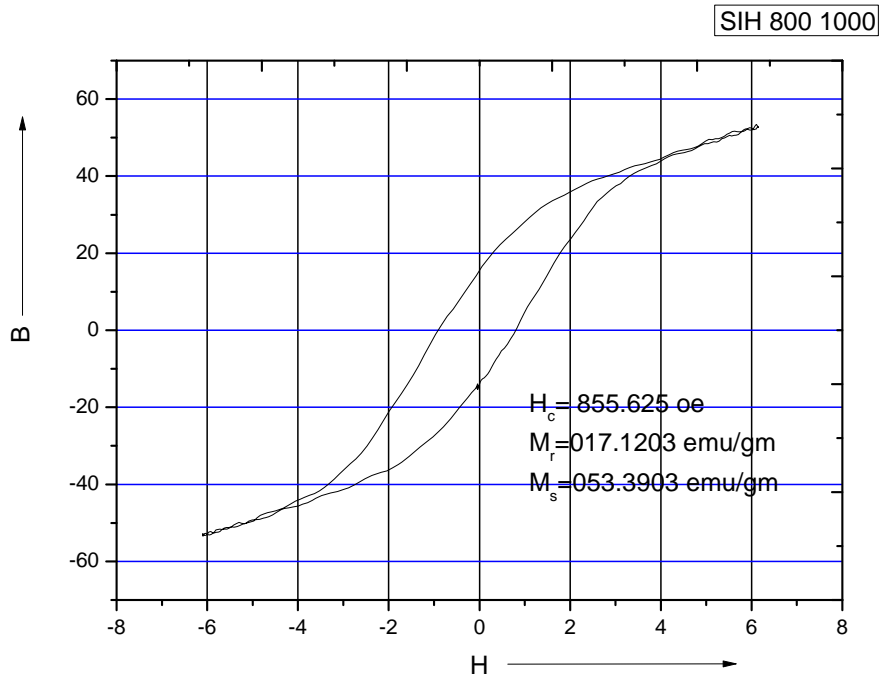


Figure 9:- B-H curve of sample SIH calcined at 800°C and sintered at 1000°C.

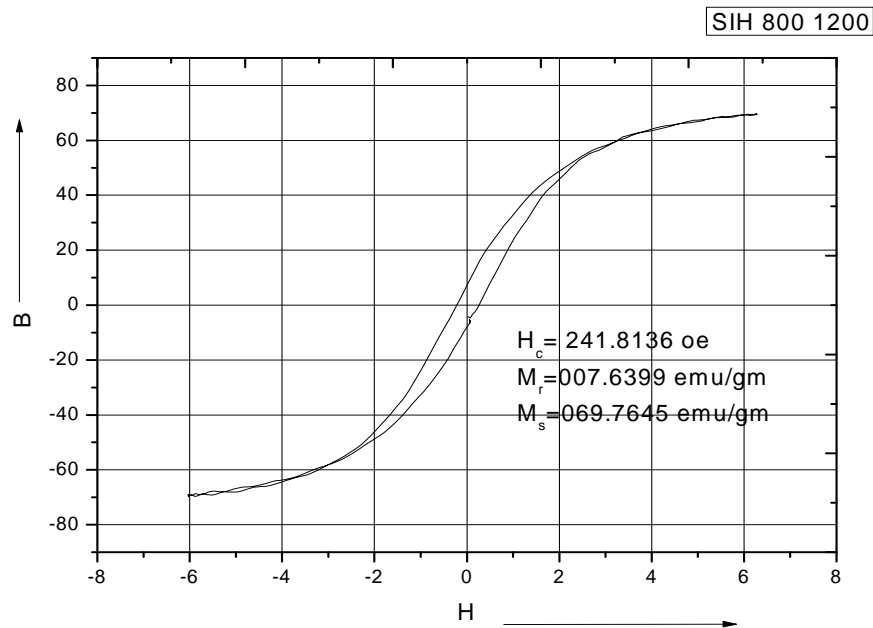


Figure 10:- B-H curve of the sample SIH calcined at 800°C and sintered at 1200°C

Form fig 9 and fig 10 it can be seen that the magnetic saturation and magnetic remenance is decreasing with the increase in sintering temperature while the magnetic saturation is increasing. the magnetic saturation value is increasing from 53 emu/gram to 69 emu/gram while the magnetic corecivity is decreasing from 855 Oe to 241 Oe.

PROCEDURE 5:

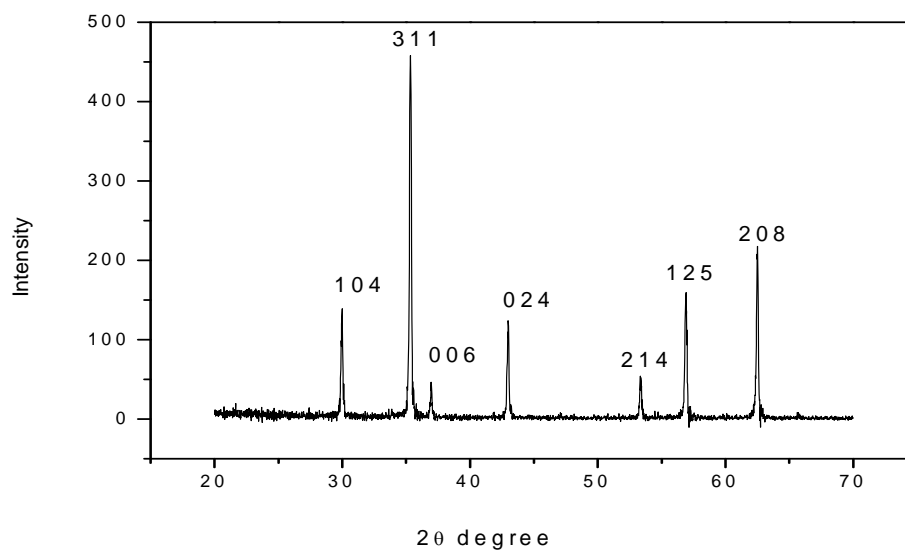


Figure 11:- XRD of sample C3 calcined at 800°C

Fig 11 shows the XRD analysis of sample C3. All the major peaks showed the cobalt ferrite phase and no other phase was present. Peak (311), peak (208) peak (104), all showed the cobalt ferrite phase.

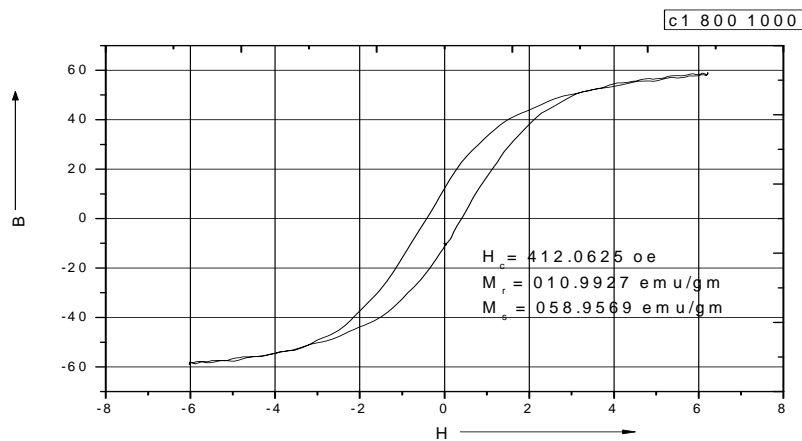


Figure 12:- B-H curve of sample C1 calcined at 800°C and sintered at 1000°C

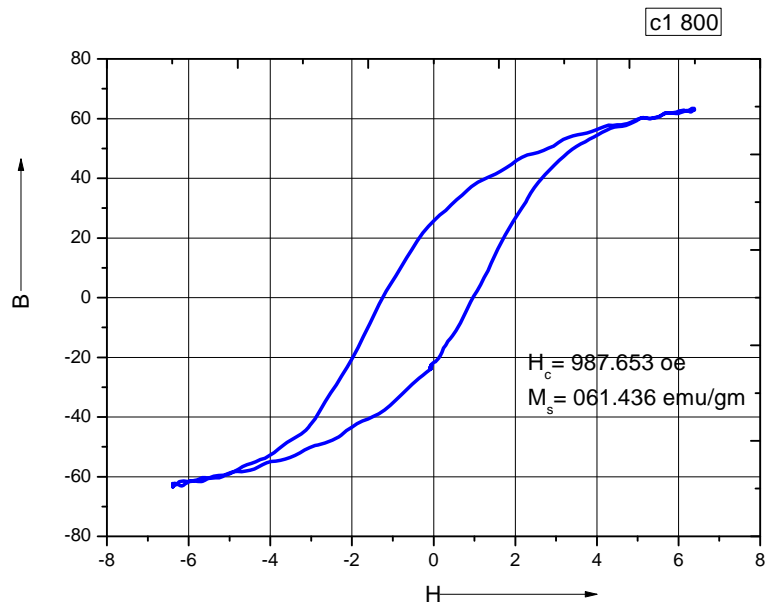


Figure 13:- B-H curve of sample C1 calcined at 800°C

From Fig. 12 and Fig. 13, it can be seen that the value of magnetization corecivity is decreasing (987 Oe to 412 Oe) and magnetic saturation is increasing with the increase in sintering temperature.

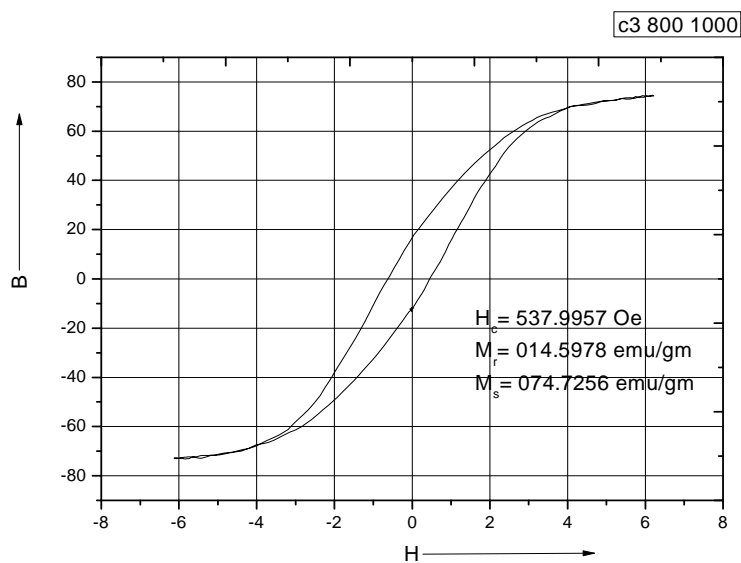


Figure 14:- B-H curve of sample C3 calcined at 800°C and sintered at 1000°C

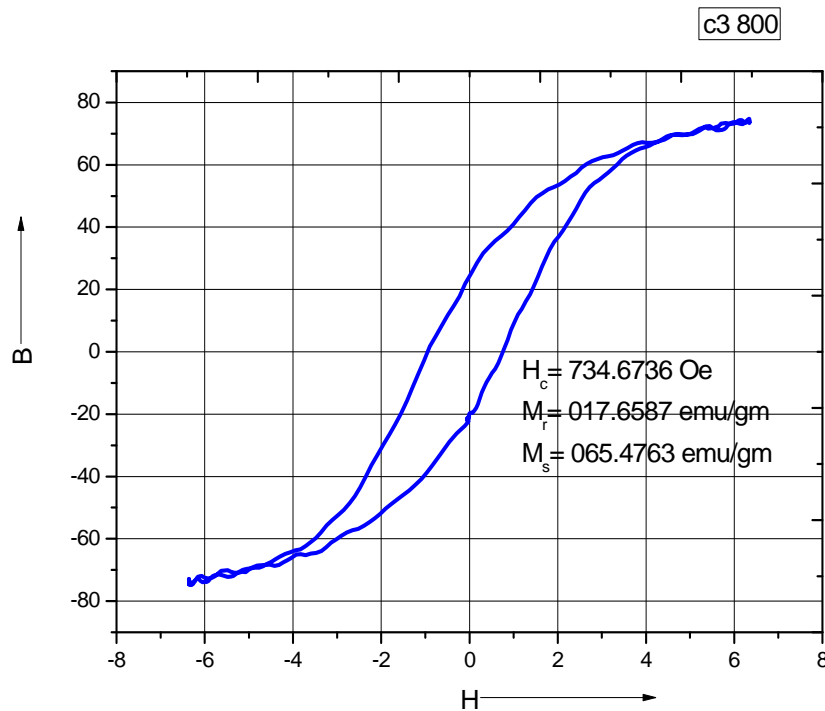


Figure 15:- B-H curve of the sample C3 calcined at 800°C

Form Fig. 15 and Fig. 16, it can be seen that the corecivity value is decreasing with the increase in the sintering temperature and the value of magnetic saturation is increasing. The value of magnetic corecivity is decreasing from 734 Oe to 537 Oe and magnetic saturation is increasing from 65 emu/gram to 74 emu/gram.

PROCEDURE 6:

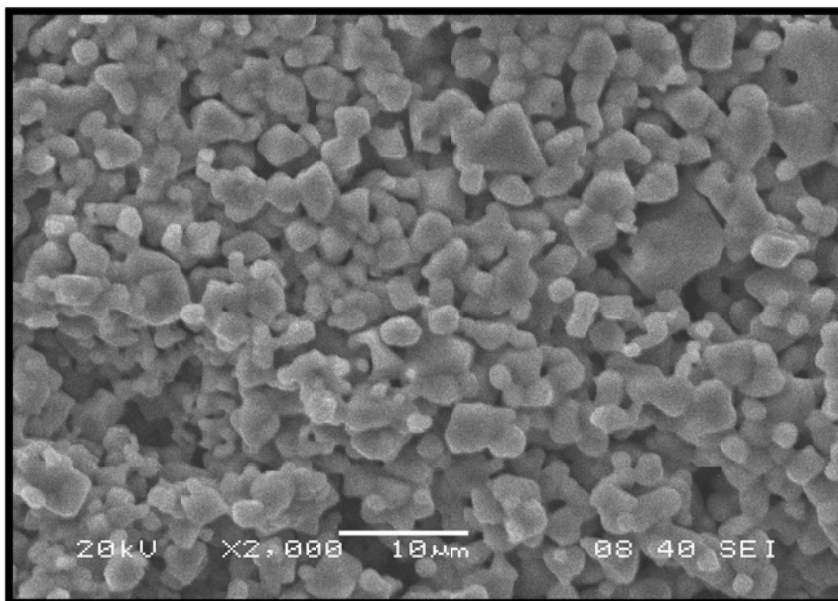


Figure 16:- SEM image of sample HISCO

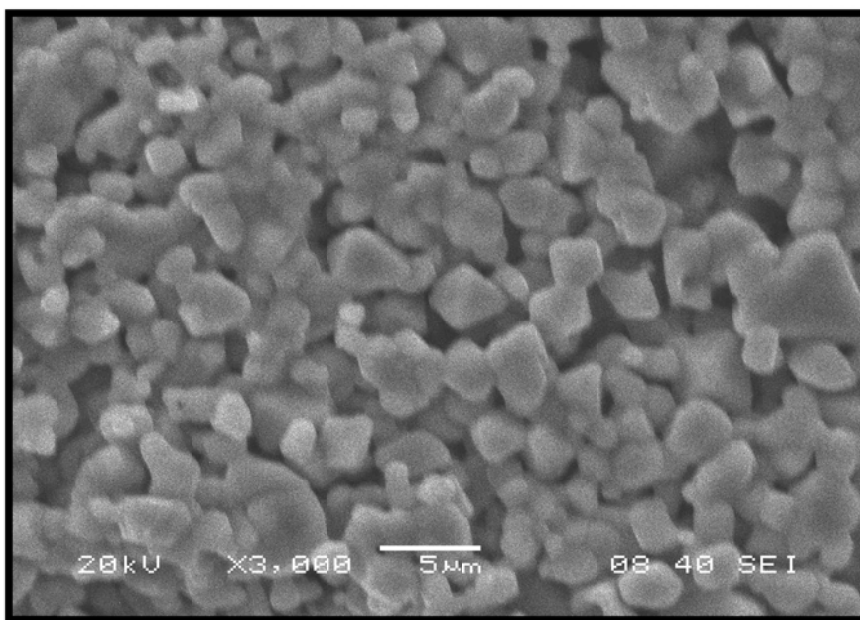


Figure 17:- SEM image of sample HISCO

Fig 16 and Fig 17 shows the SEM image of sample HISCO. It can be seen that the grains have a uniform distribution with about a circular morphology. Some amount of pores are also present in image. The average particle size of the grains is about 3 μm .

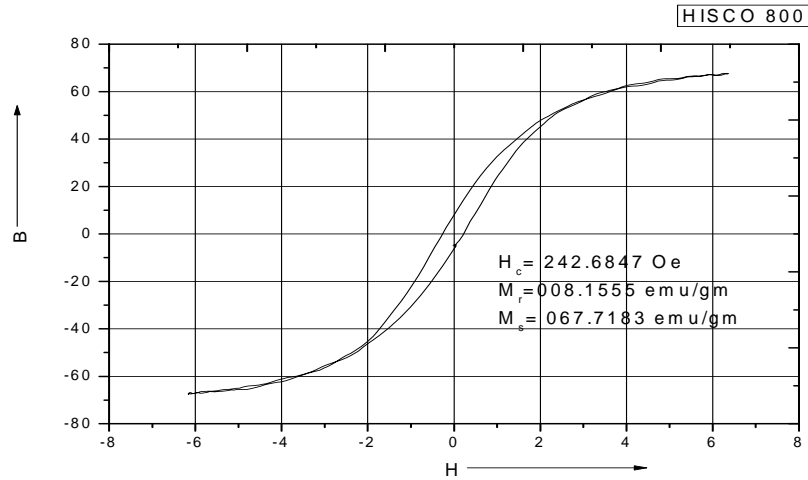


Figure 18:- B-H curve of the sample HISCO calcined at 800°C

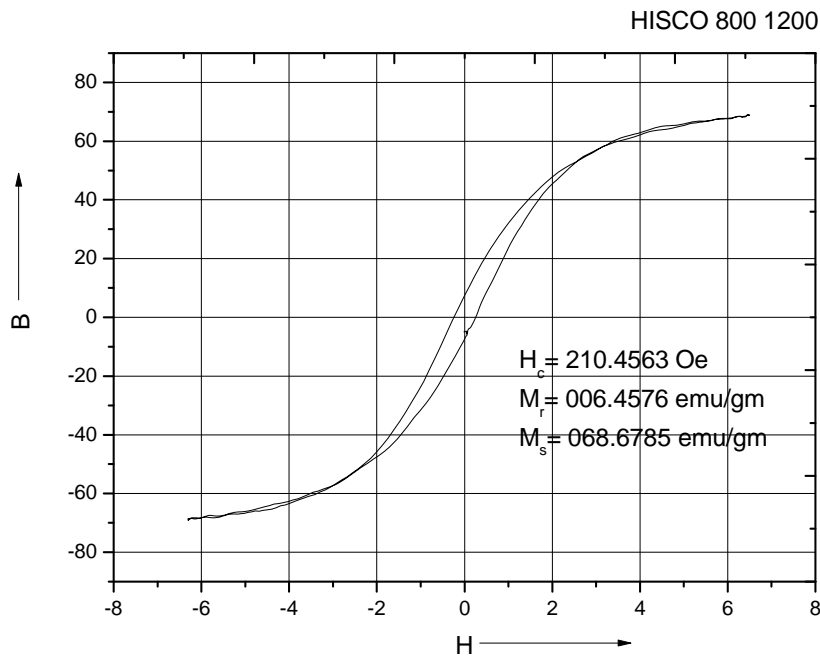


Figure 19:- B-H curve of the sample HISCO calcined at 800°C and sintered at 1200°C

Fig 18 and Fig 20 shows the B-H curve of the sample HISCO calcined at same temperature but sample in Fig 18 not sintered but sample in Fig 19 sintered at 1200°C . The graph shows that the magnetic saturation is increasing with the increase in sintered sample from 67 emu/gram to 69 emu/gram while the magnetic corecivity decreases from 242 Oe to 210 Oe.

PROCEDURE 7:

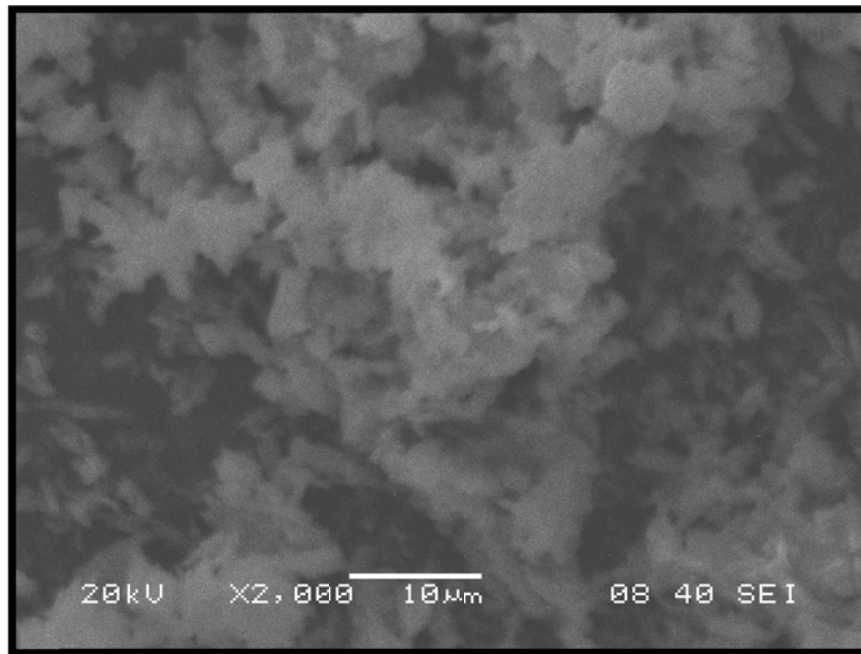


Figure 20: SEM image of sample HISG powder (low magnification)

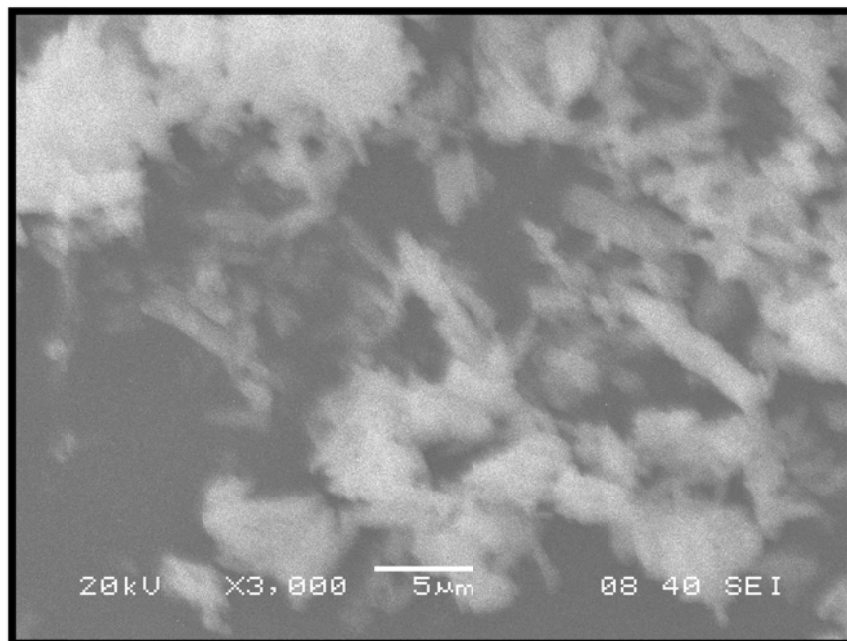


Figure 21: SEM image of sample HISG powder (high magnification)

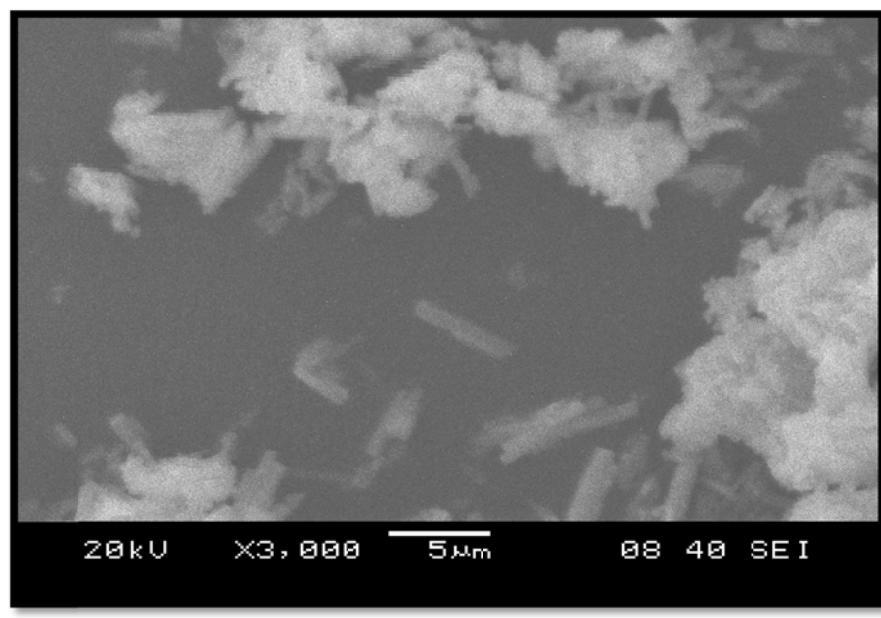


Figure 22: SEM image of sample HISG powder (high magnification)

Fig 20, Fig 21 and Fig 22 shows the SEM image of a sample powder HISG with different magnification. From the above figure it can be seen that most of the powder are in agglomerate form. Magnifying at single grains shows some of rod shape morphology

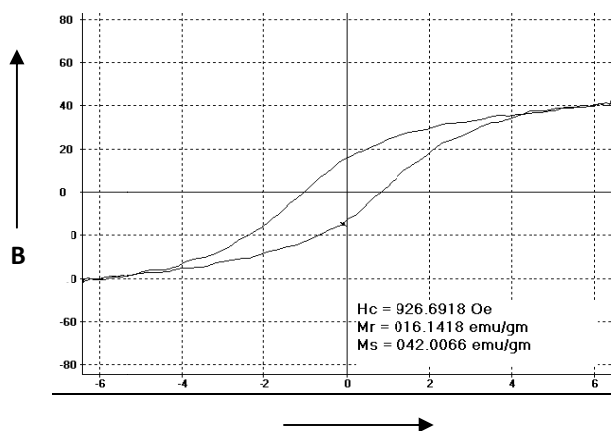


Figure 23: B-H curve of HISG calcined at 800°C

Fig 23 shows the B-H curve of the sample HISG calcined at 600°C. The magnetic corecivity was found to be very high about 926 Oe and the magnetic saturation value about 42 emu/gram.

CHAPTER 5

C CONCLUSIONS

Form the experiments it was concluded that

- Cobalt ferrite prepared from different procedure showed different morphology and particle size.
- In presence of surfactant, the sample prepared showed high magnetic corecivity compared with procedure in which no surfactant used.
- The magnetic corecivity value decreased with the increase in calcination temperature and also with the sintering temperature.
- The magnetic saturation value increased with the increase in calcination temperature and sintering temperature.
- The magnetic corecivity value also depend upon the solvent used for which it showed surfactant used in Ethylene Glycol showed better magnetic corecivity.

CHAPTER 6

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