Synthesis and Characterization of Ferrihydrite Nanoparticles

A Dissertation submitted

In Partial Fulfilment of the Requirements

For the degree of

MASTER OF SCIENCE

In

PHYSICS

BY

Neha sharma

(Roll No: 301004011)

Under the guidance of

Dr. S. D. Tiwari

SCHOOL OF PHYSICS AND MATERIALS SCIENCE

THAPAR UNIVERSITY

PATIALA-147004

INDIA

July 2012

Certificate

This to certify that the dissertation entitled "Synthesis and Characterization of Ferrihydrite Nanoparticles" submitted by Neha Sharma is in partial fulfillment for the degree of Master of Science in Physics in this university.This work has been done under my supervision. She has not submitted this material for credit towards any other degree at this or any other university.

(Dr. S.D. Tiwari)

Assistant Professor

School of Physics and material Science

Thapar university, patiala

Countersigned by

Dr. Kulvir Singh
Head

School of Physics & Material Science

Thapar University, Patiala

Dr. S.K. Mohapattra

Dean of Academic Affairs

Thapar University, Patiala

Declaration

I hereby declare that the dissertation entitled "Synthesis and Characterization of Ferrihydrite Nanoparticles" is the work carried out by me under the supervision of Dr.S.D Tiwari. I have not submitted this work anywhere else for the award of any degree

Neha Sharma

Acknowledgments

I thank my project supervisor Dr. S. D. Tiwari for his valuable guidance during the course of this work. I really appreciate him for his help, patience, discussions with me and encouragements.

I owe my sincere gratitude to my family whose support and obstinate love gave me the energy to complete this thesis work successfully and also for their untiring help during the difficult moment.

Neha sharma

Abstract

Nanoparticles of ferrihydrite are prepared by a sol gel method. The sample is characterized by x-ray diffractometer, transmission electron microscope and vibrating sample magnetometer. The average crystallite size is found to be about 6 nm. The prepared nanoparticles of ferihydrite are found to be antiferromagnetic at room temperature.

Contents

List of Figures

CHAPTER 1

Introduction

All matters are made up with atoms. Atoms have a centre known as nucleus having positive charge. A nucleus is consisting of positively charged protons and neutral neutrons. Nucleus is orbited by one or more negatively charged particles known as electrons. An electron describes its motion in two ways. It moves around nucleus and also spins along its own axis. The spin and orbital motion of an electron produce a magnetic field. The spin and orbital motion of an electron in an atom behave as current carrying loops. These loops produce magnetic fields. The direction of the magnetic field is determined by the direction of spin and orbital motion. The strength of the field is called magnetic moment. Thus the atom possesses magnetic moments in two ways. First is due to orbital motion of electron and the second is due to its spin. The total magnetic moment is the vector sum of orbital magnetic moment and spin magnetic moment [1-5].

1.1 Classification of magnetic materials

Depending on the behavior of materials in external applied magnetic field the materials can be classified into following categories [1-5].

1.1.1 Diamagnetism

Diamagnetism is a fundamental property of all matters. Diamagnetic materials contain atoms which have zero magnetic moment. The susceptibility of diamagnetic materials is always negative and the relative permeability is less than one. Examples of diamagnetic materials are copper, bismuth, sodium chloride etc.

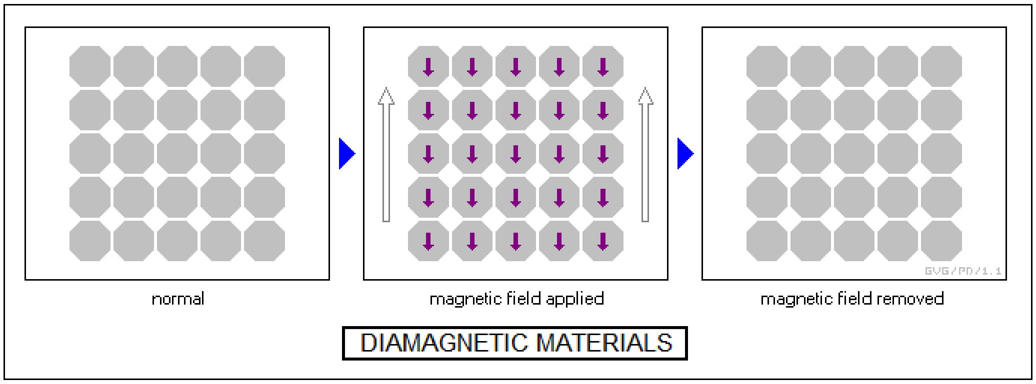

Figure 1.1: Arrangement of spins in diamagnetic materials [6].

Figure 1.2: M vs. H curve for a diamagnetic material [7].

1.1.2 Paramagnetism

In this class of materials atoms or ions of materials have some non zero net magnetic moment due to unpaired electrons. When magnetic field is applied to these types of materials the dipole moments try to align along the direction of applied field. In presence of magnetic field these materials have positive susceptibility.

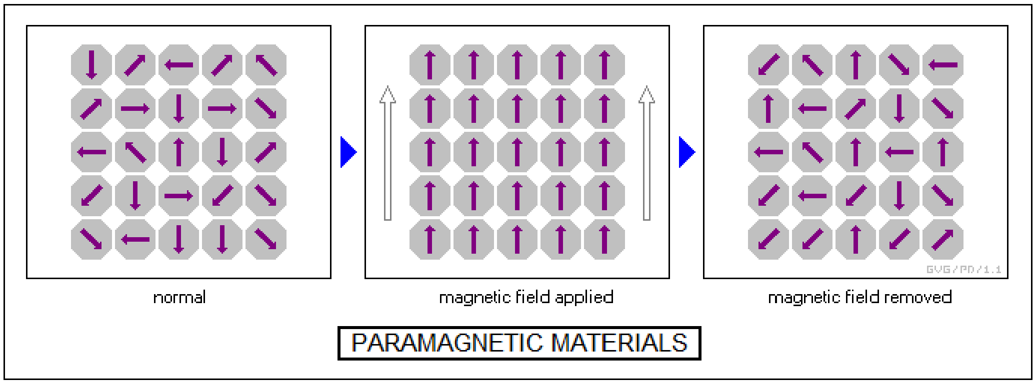

Figure 1.3: Arrangement of spins in paramagnetic materials [6].

The magnetization of these materials decreases with increasing temperature according to relation

$$M = C\left(\frac{B}{T}\right)$$

Here M is the Magnetization, C is the Curie constant which is different for different materials and T is the temperature. This relation is known as the Curie law

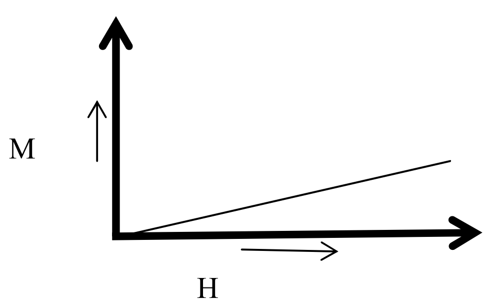

Figure 1.4: Magnetization as a function of applied field for paramagnetic materials [7].

1.1.3 Ferromagnetism

The atomic magnetic moments in these materials exhibit very strong interaction known as exchange interaction. This strong interaction is produced by electronic exchange forces and results into parallel alignment of magnetic moments. The ferromagnetic material shows spontaneous magnetization even in the absence of magnetic field. The exchange interaction is very strong but is short range in nature.

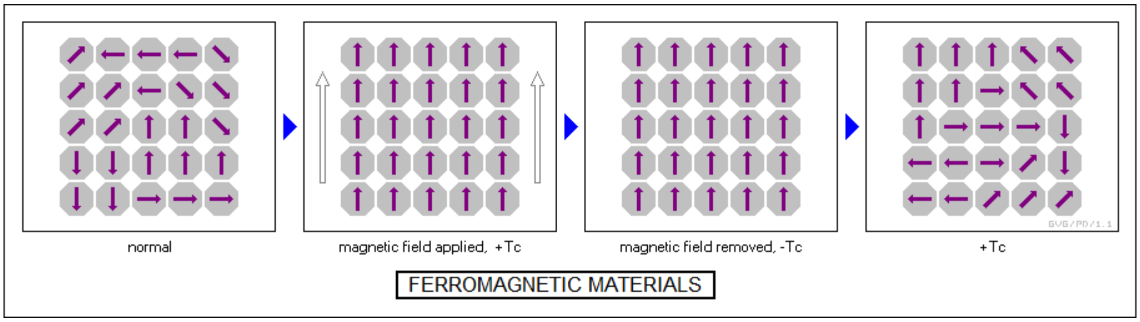

Figure 1.5: Arrangement of spins in ferromagnetic materials [6].

At a critical temperature T_C, known as the Curie temperature, a ferromagnetic material becomes paramagnetic. At this temperature the thermal energy becomes larger than the exchange interaction energy between spins. Due to this reason the ferromagnetic ordering breaks and the material becomes paramagnetic. Above the Curie temperature the susceptibility of the system can be expressed by relation

$$\chi = \frac{C}{T - T_C}$$

Here C is material dependent constant.

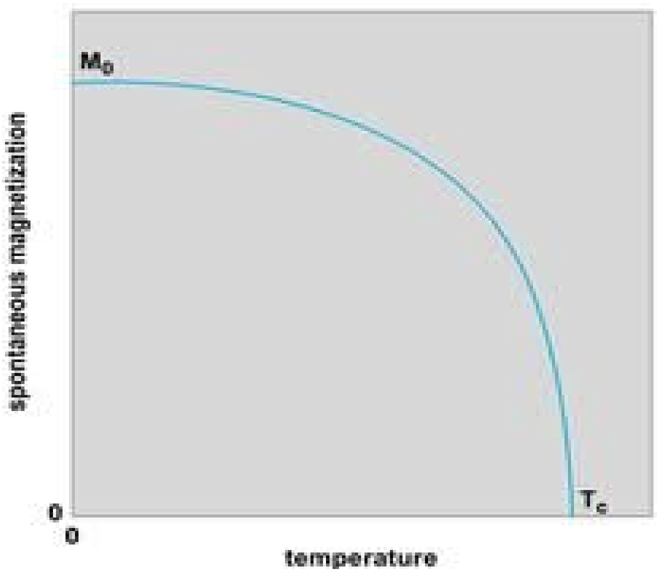

Figure 1.6: Magnetisation as a function of temperature for a ferromagnetic material [8].

1.1.4 Antiferromagnetism

In antiferromagnetic materials the spins are arranged antiparallel to each other as shown in Figure 1.7. Because of this the material posses zero net magnetization. At a critical temperature, known as the Neel temperature T_N, the antiferromagnetic ordering breaks and the material becomes paramagnetic. Well above the Neel temperature the susceptibility of the material is described by relation

$$\chi = \frac{C}{T + T_N}$$

Here C is the material dependent constant.

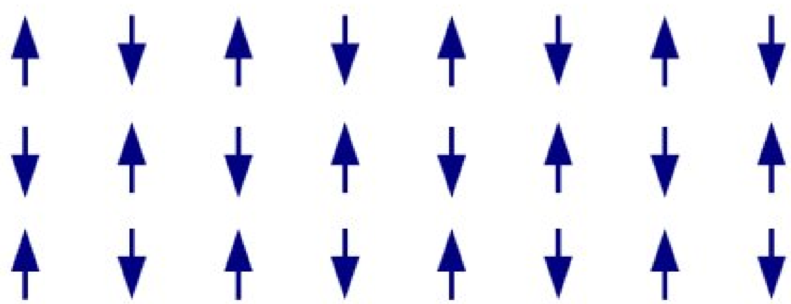

Figure 1.7: Arrangement of spins in antiferromagnetic materials [9].

1.1.5 Ferrimagnetism

In ferrimagnetic materials the spin of atoms are arranged in opposite directions. But the magnitudes of neighboring moments do not equal to each other as in the case of antiferromagnetic materials. Due to this imbalance in magnitudes of spins the material posses a non zero magnetization. At a critical temperature the ferrimagnetic ordering breaks and the material becomes paramagnetic.

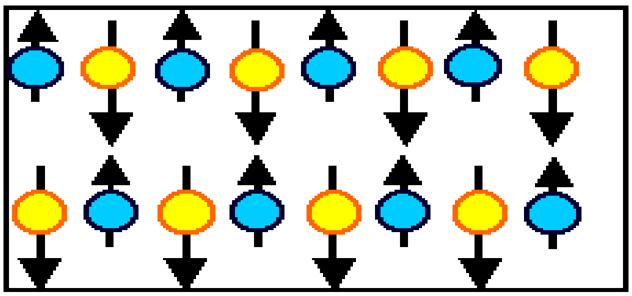

Figure 1.8: Arrangement of spins in ferrimagnetic materials [10].

CHAPTER 2

Experimental Details

2.1 Synthesis

In this work, nanoparticles of ferrihydrite are synthesized by a sol gel route [11]. The nanoparticles are prepared by reacting aqueous solution of ferric nitrate and sodium hydroxide. For this sodium hydroxide solution is added drop wise into aqueous solution of ferric nitrate with constant stirring until pH of the system reaches to 10. The resulting precipitate is filtered and washed several times. The precipitate is dried at 65 ^{0}C.

2.2 Characterization Techniques

2.2.1 X-Ray Diffraction

This is an important technique for the structural characterization of any material. In this a beam of x-ray undergoes Bragg's diffraction as shown in Figure 2.1. The condition for maximum in the intensity of diffracted beam is given by relation [1-5]

$$2\, d \,\text{Sin}\, \theta = n\, \lambda,$$

where d is interplaner spacing and λ is the wavelength of x-ray used. Here n = 1, 2…. This relation is known as Bragg's law.

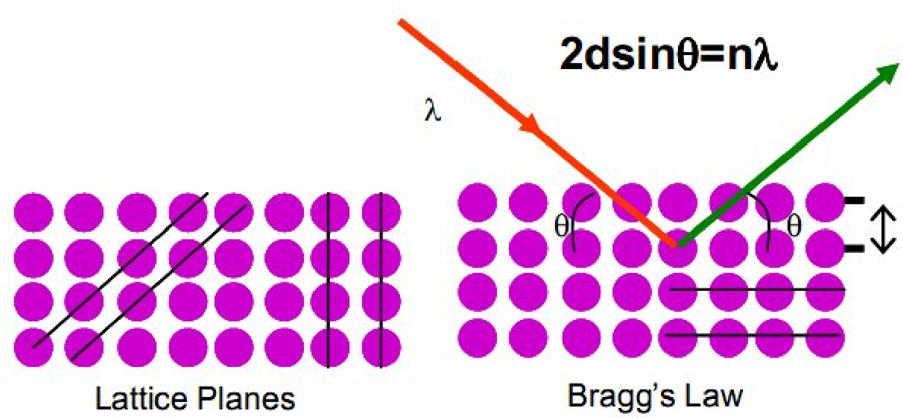

Figure 2.1: Diffraction of x-ray beam [12].

2.2.2 Transmission electron microscope

Microscope is used to get a magnified view of an object. In transmission electron microscope a beam of energetic electrons is transmitted through a thin object to get a magnified view of the object. This technique gives information about the morphology, crystal structure, defects, phases etc. of the specimen [13].

2.2.3 Vibrating Sample Magnetometer

Vibrating sample magnetometer is used to measure the magnetization of given sample. It is based on the principle of Faraday's law of induction. This law tells that changing magnetic field indices an electrical field. By measuring the induced electric field we can find the changing magnetic field.

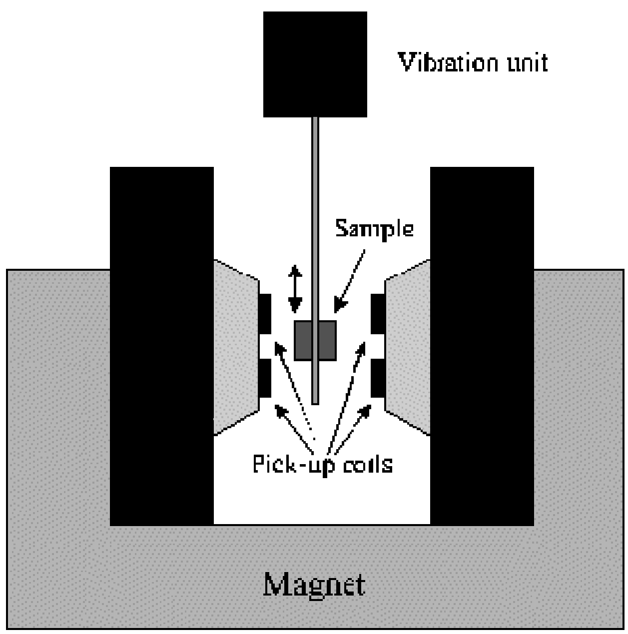

Figure 2.2: Vibrating Sample Magnetometer [14].

In this instrument an electromagnet is used to magnetize the sample. This magnetized sample is allowed to vibrate along vertical direction inside a coil. Due to the vibration of the magnetized sample the magnetic field inside the coil changes with time. This changing magnetic field produces an electric field across the coil. This field is measured using appropriate techniques and the magnetization of the sample is estimated with the help of proper calibration.

CHAPTER 3

Results and Discussions

3.1 Structural Characterization

Room temperature x-ray diffraction pattern of the prepared powder sample is shown in Figure 3.1. This figure shows that the sample is poorly crystallized. This x-ray diffraction pattern is somewhat similar to shown by others [11]. This pattern also shows that the peaks are broadened. This indicates that the material is nano crystalline. The average crystallite size is calculated using the modified Scherrer formula

$$d = 0.9\ \lambda / \cos\theta_B\ (B_M - B_S)$$

where λ is the wavelength of the x-ray used, θ_B is the Bragg angle, B_M is the full width at half-maximum (FWHM) of a peak and B_S is the FWHM of the same peak of a standard sample. The use of $(B_M - B_S)$ instead of B_M in the Scherrer formula takes care of instrumental broadening. The average crystalline size using the modified Scherrer formula turns out to be about 6 nm.

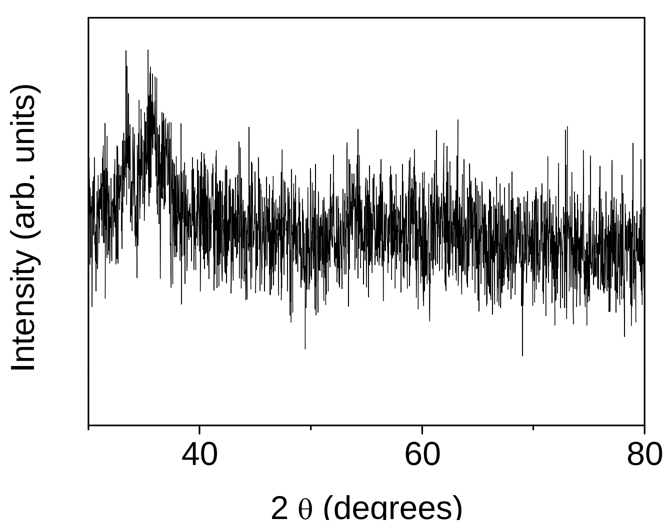

Figure 3.1: Room temperature x-ray diffraction pattern of ferrihydrite nanoparticles.

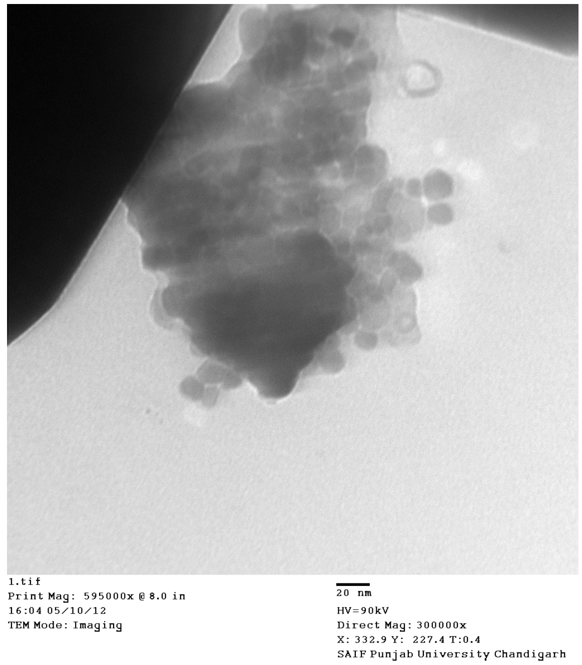

Figure 3.2: Transmission electron micrograph of ferrihydrite nanoparticles.

The prepared sample of ferrihydrite nanoparticles is also characterized by transmission electron microscope. For this the prepared powder sample is dispersed in methyl alcohol and a drop of this dispersion is allowed to dry on a transmission electron microscopy grid. The micrograph thus obtained is shown in Figure 3.2.

3.2 Magnetization Measurement

Magnetization as a function of magnetic field measurement is done for ferrihydrite nanoparticles at room temperature. This is shown in Figure 3.3. This figure shows that there is no hysteresis in the magnetization as a function of magnetic field curve. The magnetization of the sample increases with increasing magnetic field linearly at higher magnetic fields but the same increases nonlinearly at lower magnetic fields. These are characteristics of antiferromagnetic material. Also there is no sign of saturation of the magnetizations for this sample because antiferromagnetic materials usually require very high magnetic field to saturate.

.

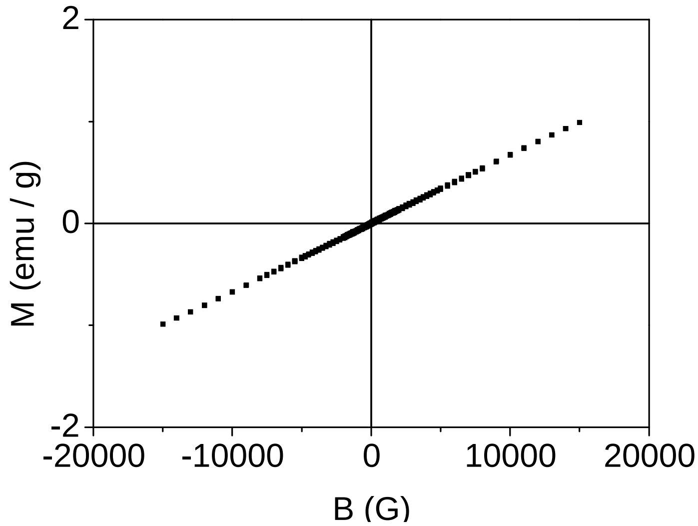

Figure 3.3: Magnetization as a function of applied magnetic field at room temperature for ferrihydrite nanoparticles.

Magnetic susceptibility is defined as ratio of the magnetization to the applied magnetic field. For this the linear portion of the magnetization versus magnetic field curve at higher magnetic field is used. The magnetic susceptibilities of the sample is found to about 6.6×10^{-5} emu / g Oe.

CHAPTER 4

Conclusions

In this work ferrihydrite nanoparticles are synthesized by a sol gel method. The sample is characterized by x-ray diffraction, transmission electron microscope and vibrating sample magnetometer. The sample is found to be poorly crystallized. The average crystallite size is found to be about 6 nm. The prepared sample of ferrihydrite nanoparticles is also found to be antiferromagnetic at room temperature.

In this work the behaviour of 6 nm ferrihydrite particles is studied. One may further synthesis ferrihydrite nanoparticles of different sizes and can study their behavior in details.

References

1. C. Kittel, *Introduction to Solid State Physics* (John Wiley and Sons, Inc., Seventh Edition, 1996).
2. A. J. Dekker, *Solid State Physics* (Macmillan India Limited 1996).
3. Ashcroft N.W. and Mermin N.D., *Solid State Physics* (Harcourt Asia Pvt. Ltd., 1976.
4. V. Raghavan, *Materials Science and Engineering* (PHI Learning Private Limited, Fifth Edition, 2010).
5. W. F. Smith and J. Hashemi, *Foundation of Materials Science and Engineering* (McGraw Hill International Edition, Fourth Edition, 2006).
6. http://www.vectorsite.net/tpqm_04.html
7. Ajay Kumar, M. Sc. Project, Thapar University, 2011.
8. http://accessscience.com/content/Curie%20temperature/173700
9. http://en.wikipedia.org/wiki/File:Antiferromagnetic_ordering.svg
10. http://www.tf.uni-kiel.de/matwis/amat/elmat_en/kap_4/backbone/r4_1_3.html
11. M. S. Seehra, V. S. Babu, A. Manivannan and J. W. Lynn, Phys. Rev. B **61**, 3513 (2000).
12. http://www.asdlib.org/onlineArticles/ecourseware/Bullen_XRD/LearningActivity_Diffraction_BraggsLaw.pdf)
13. http://en.wikipedia.org/wiki/Transmission_electron_microscopy
14. http://www.el.utwente.nl/tdm/istg/research/vsm/vsm.htm