

Synthesis and Characterization of Alpha Nickel Hydroxide Nanoparticles

A

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Under the guidance of

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INDIA

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*Dedicated to my
family*

CERTIFICATE

This is to certify that the project report entitled "Synthesis and Characterization of Alpha Nickel Hydroxide Nanoparticles" submitted by Ms. Priya is in partial fulfilment for 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.



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DECLARATION

I hereby declare that the project report entitled "Synthesis and Characterization of Alpha Nickel Hydroxide Nanoparticles" is the work carried out by me under the supervision of Dr. S. D. Tiwari. The matter embodied in this dissertation has not been submitted anywhere else for the award of any other degree.

Priya
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ABSTRACT

Alpha Nickel Hydroxide nanoparticles are synthesized by a chemical route. The sample is characterized by x-ray diffraction, thermogravimetric analysis and vibrating sample magnetometer. The average crystallite size is found to be about 6 nm. The prepared nanoparticles of α -Ni(OH)₂ are found to be paramagnetic at room temperature.

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CHAPTER 1

INTRODUCTION

Motions of electric charges inside materials result in forces of attraction and repulsion between the materials. Magnetic behaviour of any material is due to orbital motion and spin of electrons in the atoms. This is also due to how the electrons interact with each other. Depending on the behaviour of materials in external applied magnetic field all materials can be classified in several categories [1-3].

Diamagnetism is a fundamental property of all materials. Diamagnetic materials contain atoms which do not have any unpaired electrons. The susceptibility of diamagnetic materials is always negative. Also the relative permeability of diamagnetic materials is less than one. Examples of such materials are copper, bismuth, sodium chloride etc.

In paramagnetic materials the atoms or ions have a net magnetic moment due to unpaired electrons in partially filled orbitals. These moments are randomly oriented. So in the absence of external applied magnetic field, these materials do not have any net magnetic moment. When an external magnetic field is applied, the moments try to align themselves along the direction of the applied field. The susceptibility for these materials is positive and small. The susceptibility of paramagnetic materials decreases with increasing temperature according to Curie law.

The atomic or ionic magnetic moments in ferromagnetic materials exhibit very strong interaction. This interaction is known as exchange interaction. This strong interaction is produced by electronic exchange forces. Due to this the moments are aligned in same direction. The ferromagnetic material shows spontaneous magnetization in the absence of magnetic field. The exchange interaction is very strong but is short range in nature. 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. Due to this reason the ferromagnetic ordering breaks and the material becomes paramagnetic above this critical temperature.

Antiferromagnetic materials possess a magnetic ordering in which the magnetic moments of alternating atoms or ions in the crystal are aligned in opposite directions. Antiferromagnetic materials have a small but positive susceptibility. They do not possess any magnetic moment in absence of magnetic field and at lower temperature. Antiferromagnetism

occurs only below a critical temperature called Néel temperature. Above Néel temperature the antiferromagnetic ordering breaks and the materials become paramagnetic.

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

1.1 Nickel Hydroxide

Nickel hydroxide is known to be a technologically important material. Its most important application is in rechargeable battery systems. This material exists in two phases i.e. alpha and beta phases [4]. The α -Ni(OH)₂ phase is relatively disordered with larger interlayer spacing. This spacing accommodates anions like nitrate, carbonate, sulfate as well as water molecules. The hexagonal β -Ni(OH)₂ phase is brucite-like with properly oriented Ni(OH)₂ layers. These layers are perfectly stacked along the c-axis.

1.2 Literature Review

A brief review of recent works on alpha nickel hydroxide is as below.

Jeevanandam et al. (2001): It was reported that nanosized α -Ni(OH)₂ synthesised by ultrasound radiation shows good stability in alkaline medium like KOH [5].

Hu et al. (2003): Studied Al-substituted α -Ni(OH)₂. It was noticed that with increase in Al contents the electrochemical discharge capacity decreases. It was found that nickel hydroxide with 10% Al content has satisfactory stability in strong alkali medium [6].

Ramesh et al. (2008): They reported the temperature dependent synthesis of alpha and beta Nickel hydroxide [7].

Tiwari et al. (2008): They studied the variation of magnetization with temperature and found that at higher temperature Ni(OH)₂ is paramagnetic and at lower temperature there is transition from paramagnetic to ferromagnetic and further decrease in temperature results in superparamagnetic blocking [8].

Rall et al. (2010): They compared the properties of α and β Ni(OH)₂ and reported the presence of metamagnetism in beta nickel hydroxide whereas alpha nickel hydroxide is hard ferromagnet below their respective ordering temperature[4].

Liu et al. (2010): They found transition from paramagnetic to antiferromagnetic in nickel hydroxide nanoparticles at low temperature [9].

Rall et al. (2011): They studied the magnetic properties of alpha nickel hydroxide and found the ferromagnetic ordering in the synthesised sample [10].

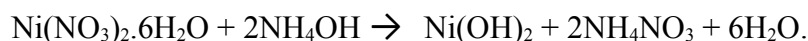
CHAPTER 2

EXPERIMENTAL DETAILS

2.1 Synthesis

There are various physical and chemical methods for the preparation of nanocrystalline materials. In the present work alpha nickel hydroxide was synthesized via chemical route.

The α -Ni(OH)₂ is prepared by adding 2M of ammonium hydroxide in 1M of nickel nitrate hexahydrate solution at 4 °C [7]. The ammonium hydroxide solution is added drop wise under constant stirring into the nickel nitrate hexahydrate solution till pH 10 is achieved. The chemical reaction between aqueous nickel nitrate hexahydrate and ammonium hydroxide is as follows:



The precipitate thus formed is aged in the solution for 15 hours. After that the precipitate is washed several times with distilled water. Finally the green colour precipitate is dried at 60 °C and then grinded to get fine powder sample.

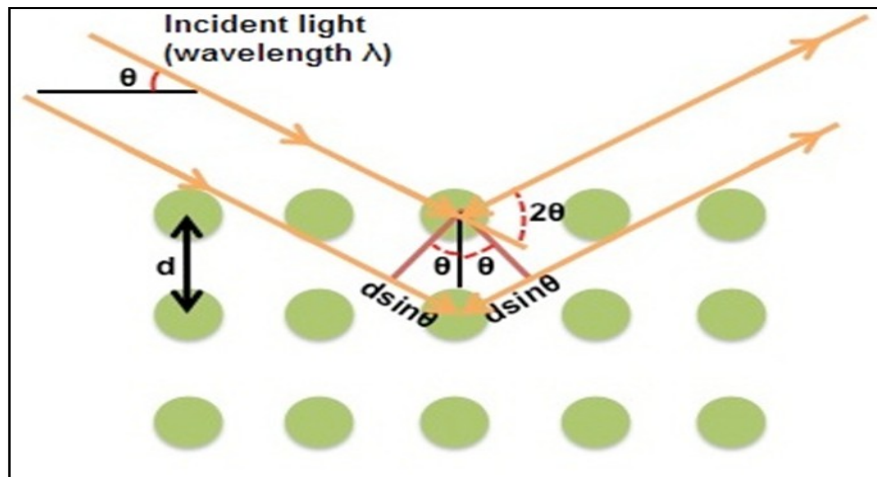
2.2 Experimental Techniques

2.2.1 X-ray Diffraction

This technique is used for structural characterization of materials. In this a beam of x-ray undergoes Bragg's diffraction. The condition for maximum in the intensity of diffracted beam is given by relation [1-3]

$$2 d \sin \theta = n \lambda,$$

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



2.1: A beam of x-rays falling on a set of parallel atomic planes at an angle θ

The x-ray diffraction pattern of a powder sample gives plot of intensities of diffraction peaks as a function of the angle 2θ . This plot is used to calculate possible values of d and thus the given material is characterized.

2.2.2 Thermal Analysis

Many materials are not thermally stable and decompose to other materials on heating. A thermogravimetric analyser is used to check the thermal stability of a material. In this the mass of sample is measured as a function of temperature. Any change in mass of sample indicates for the change in phase of the material. A schematic diagram of thermo gravimetric analyser is shown in following figure.

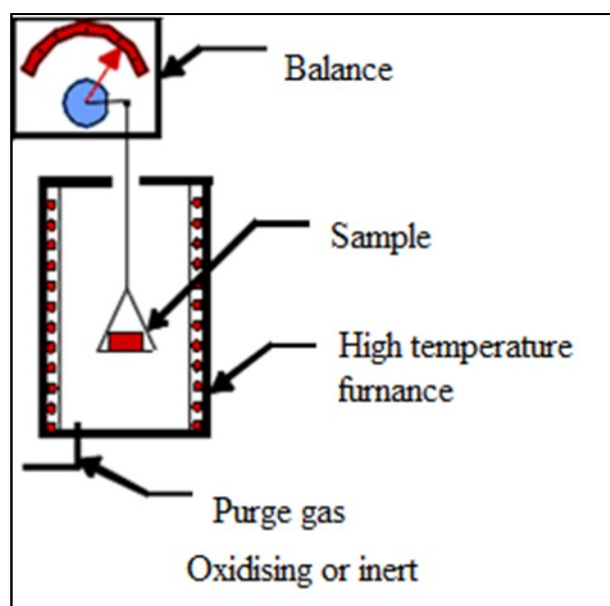


Figure 2.2: Schematic diagram of thermal gravimetric analysis [12].

2.2.3 Vibrating Sample Magnetometer

A vibrating sample magnetometer is used to measure magnetization of a sample. It is based on the principle of Faraday's law of induction. This law tells that changing magnetic flux induces an emf. This induced emf is directly proportional to the magnetization of the sample. This instrument uses an electromagnet 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 emf across the coil. This emf is measured using appropriate techniques and the magnetization of the sample is estimated with the help of proper calibration.

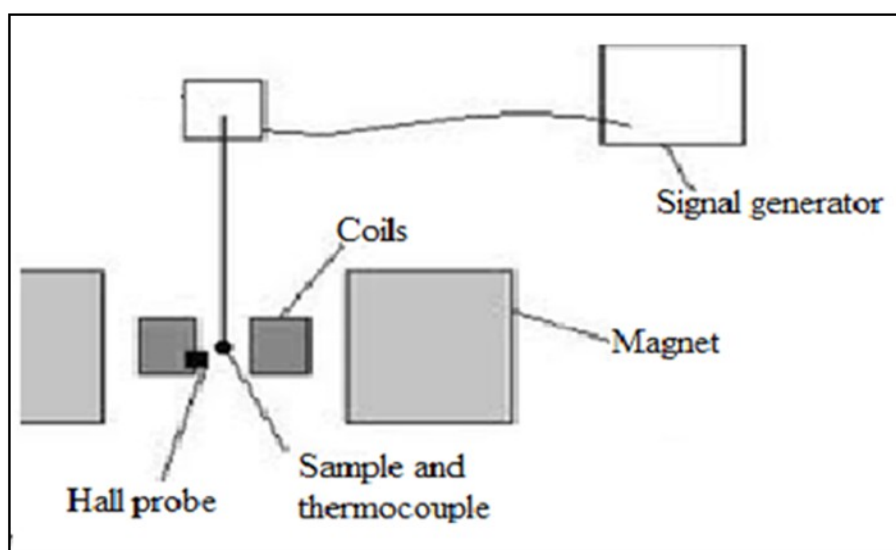


Figure 2.3: Schematic diagram of vibrating sample magnetometer [13].

CHAPTER 3

RESULTS AND DISCUSSION

3.1 Structural Characterization

Structural characterization of prepared sample is done using x-ray diffractometer. Intensity of diffracted beam is recorded as a function of angle 2θ . The x-ray diffracted pattern of prepared green coloured powder sample at room temperature is shown in figure 3.1. From this pattern it is found that prepared green coloured powder sample is α -Ni(OH)₂.

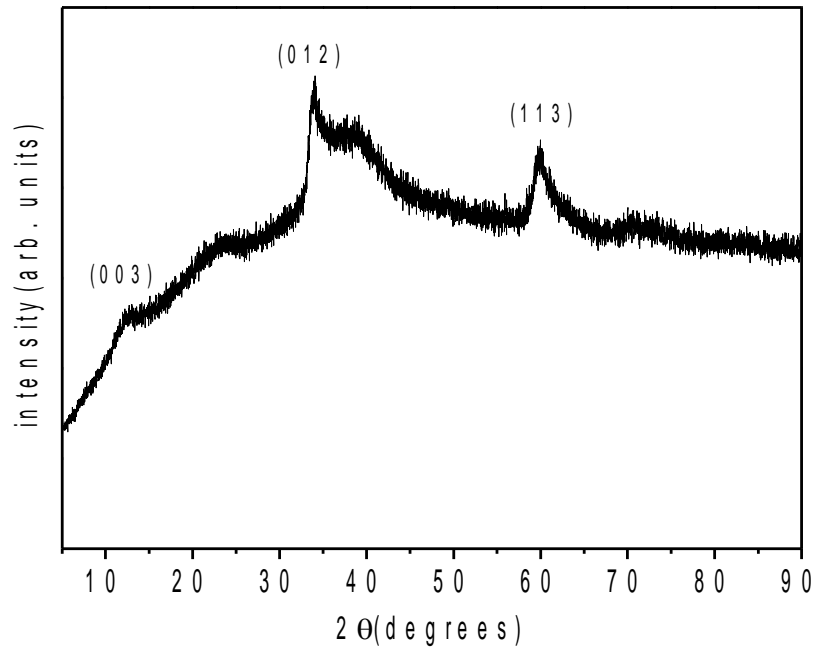


Figure 3.1: Room temperature x-ray diffraction pattern of alpha nickel hydroxide.

From figure 3.1 it is also noticed that the entire peaks are broadened. This indicates that the sample is nanocrystalline. The average crystal size is calculated using modified Scherrer formula [14].

$$d = \frac{0.9\lambda}{\cos\theta_b \sqrt{B_m^2 - B_s^2}}$$

Here d is mean crystallite size, λ is the wavelength of x-rays, θ_b is Bragg angle, B_m and B_s (in radians) is full width at half maximum of the sample and a standard sample respectively. The crystallite size of α -Ni(OH)₂ comes out to be about 6 nm.

3.2 Thermal Characterization

Figure 3.2 shows variation in the mass of sample as a function of temperature. For this the sample is heated at a constant heating rate of 10 degrees per minute in air. We see that the mass of sample suddenly starts decreasing at about 225°C. This loss in weight of the sample is due to decomposition of material into NiO.

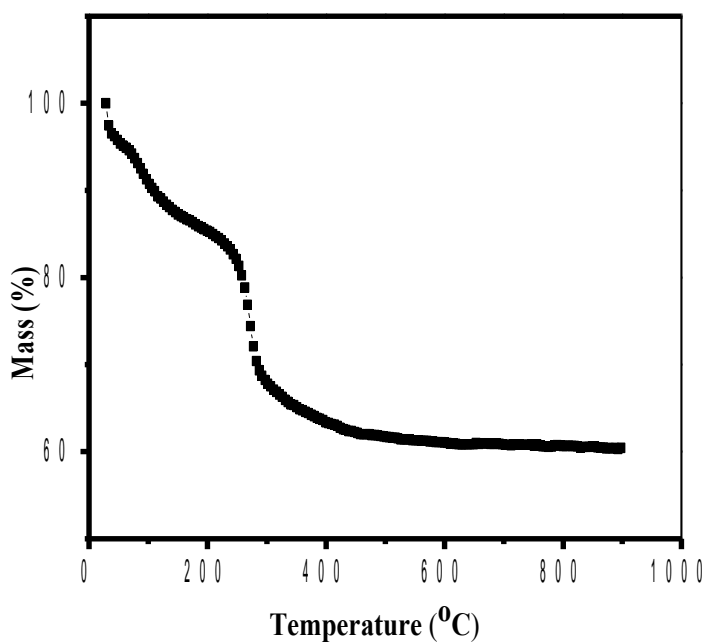


Figure 3.2: Thermogravimetric curve for alpha nickel hydroxide.

3.3 Magnetization Measurement

Magnetization as a function of applied magnetic field, at room temperature, is measured for α -Ni(OH)₂ nanoparticles using vibrating sample magnetometer. The result is shown in figure 3.3. From this curve it is found that the magnetization changes linearly with applied magnetic field. This is behaviour of paramagnetic materials. In other words the prepared sample of alpha nickel hydroxide nanoparticles is paramagnetic at room temperature.

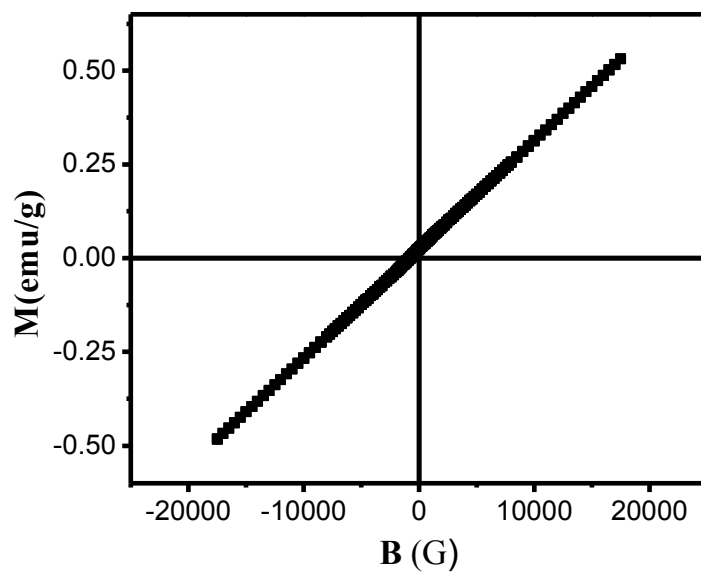


Figure 3.3: Magnetization as a function of applied magnetic field for alpha nickel hydroxide nanoparticles at room temperature.

CHAPTER 4

CONCLUSION

In this work α -Ni(OH)₂ nanoparticles are synthesized via a chemical route. The average crystallite size of the sample turns out to be about 6 nm. The sample decomposes to nickel oxide on heating in air above 225 °C. The sample is found to be paramagnetic at room temperature.

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