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Analysis And Investigation of Nano-Crystalline Hexaferrite Magnetic Nanoparticles with A Single Domain

Asha J K [1]

Research Scholar, Department of Physics,
Dr. A. P. J. Abdul Kalam University, Indore, MP, India.
Email: ashagurumurthyg@gmail.com

Dr. OM Prakash Choudhary [2]

Research Guide, Department of Physics,
Dr. A. P. J. Abdul Kalam University, Indore, MP, India.

ABSTRACT

Nanoparticle systems are the focus of this dissertation and related research. This study delves into the capabilities of magnetite, cobalt, and ferrite nanoparticle systems in terms of the magnetocaloric effect (MCE), as well as their use in polymer composites. There are several transdisciplinary realms where both fields could be useful. Using the sol-gel process in an air atmosphere at 750°C for 6 hours, the ferrite materials NiCr0.07Fe1.93O4 and NiCo0.07Fe1.93O4 were successfully synthesized. Electron spin resonance (ESR) spectroscopy, scanning electron microscopy (SEM), X-ray powder diffraction (XRD), and electrical and dielectric properties are investigated in both materials. The synthesised samples' X-ray diffraction patterns confirm the formation of a spinel structure in the Fd3m space group, free from impurities and secondary phases. The sizes of the crystallites are calculated using the Scherrer equation, starting from the most intense peak. The crystal size of NiCr0.07Fe1.93O4 is 48.5 nm, which is larger than that of NiCo0.07Fe1.93O4 (46.3 nm). The results of the experiments show that as the frequency increases, the dielectric constant ε' decreases. However, as the frequency increases, the electrical conductivity of the alternating current σac also increases.

Keywords: *Nano-Crystalline Single Domain, Hexaferrite, Magnetic Nanoparticles.*

1. INTRODUCTION

Nanoparticles can contain hundreds to thousands of atoms, depending on their size. When subjected to a magnetic field, a substance can alter its magnetic characteristics. The five most common metals are iron, diamagnetic steel, paramagnetic steel, and antiferromagnetic steel. Atoms with one unpaired electron display a net magnetic moment in ferromagnetic materials like iron, nickel, and cobalt. There are several atoms in the material's domains, and their net domain magnetic moments are all facing in the same direction. The scenario does not include a net magnetic moment since the distribution of the domains' magnetic moments is random. The domain magnetic moments of ferromagnetic materials align with the field direction when they are exposed to a magnetic field, leading to a significant net magnetic moment.

External magnetic fields have biological uses, like hyperthermia and medication targeting, since the magnetic core may selectively separate these particles for analytical reasons. Here is a rundown of all the synthetic methods that have been investigated for immobilizing polyzwitterions on magnetic nanoparticle surfaces in the last ten years.

1.1 Significance of the Study

Research into single-domain nano-crystalline hexaferrite magnetic nanoparticles is highly important because of the vast array of possible technical and industrial uses for these unusual particles' structural, functional, and magnetic characteristics. Ideal candidates for applications in high-density data storage, sophisticated biomedical technologies, and electromagnetic wave absorption are hexaferrite nanoparticles, especially in their single-domain form, which display high coercivity, great thermal stability, and exceptional magnetism. Gaining a comprehensive understanding of these nanoparticles' production, characterisation, and functional performance is essential for realizing their maximum practical potential.

Due to their single-domain structure, these nanoparticles exhibit enhanced stability and storage performance with negligible domain wall movement. Findings from this research can inform the design of more efficient and capacitive magnetic storage medium for the future. Potential biological uses for hexaferrite magnetic nanoparticles include heat therapy for cancer, tailored medication administration, and magnetic resonance imaging (MRI). By enhancing their biocompatibility and magnetic anisotropy, these nanoparticles can be modified for targeted drug delivery, enabling the exact and regulated release of medicinal substances. In addition, they have the ability to enhance MRI contrast, which can lead to more accurate diagnoses, and they have the ability to heat cancer cells locally, which can reduce harm to healthy tissues during hyperthermia treatments. This study plays a crucial role in refining these nanoparticles for use in biomedicine, making sure they work well and are safe for patients.

1.2 Magnetic Nanoparticles

In the past few years, a great deal of effort has gone into creating MNPs, studying their behavior, and finding ways to use them in biological research. Hyperthermia, biosensing, tissue healing, MRI, biochemical separations, bioanalysis, and magnetic medication targeting are just a few of the many possible applications that have prompted research on MNPs. These and related physiochemical and structural features of MNPs have been the subject of a great deal of research. Due to the significant relationship between nanoparticle size and their physicochemical characteristics, producing nanoparticles of consistent size is an important goal in the field.

Magnetic properties such as isotropy, coercivity, magnetization, blocking temperature, and more can be observed in 100 nm-sized nanoparticles of both hard and soft magnetic materials, in contrast to their bulk counterparts. One noteworthy size-dependent feature of magnetic particles is their increased coercivity in the nanoscale domain with decreasing particle size. This is the rationale behind the term "superparamagnetic" when describing particles whose coercivity is eliminated due to their extremely small size. This is why tiny magnetic particles with a high coercivity are important; low or zero coercivity is undesirable in many technical applications, such high-density data storage. There are several materials that can be used to create magnetic nanoparticles. These include iron oxides (Fe304 and y-Fe203), pure metals (Fe and Co), spinel-type ferromagnets (MgFe204, MnFe204, and CoFe204), and alloys (CoPt3 and FePt).

Excellent magnetic nanoparticles are made using a variety of techniques, such as co-precipitation, hydrothermal synthesis, micelle synthesis, thermal breakdown and reduction, and laser pyrolysis. Because of how easy they are to make in large quantities, these products have relied heavily on chemical syntheses. Nanoparticle nucleation and growth rate management is one approach to tuning magnetic characteristics. Everyone knows that in order to make monodisperse particles, there has to be a controlled development phase after a brief burst of nucleation.

1.3 Physical Characteristics of Nanomagnets

The motion of mass-charged particles generates a magnetic field. In this category you can find ions, protons, electron holes, and electrons. When a charged particle spins, it produces a magnetic dipole, which is another name for a magneton. Clustering of magnetons is a feature of ferromagnetic materials. Within a magnetic domain, all of the magnetons are aligned in the same direction due to the exchange forces. This substance is ferromagnetic. The concept of domains differentiates ferromagnetism from paramagnetism. The domain structure's magnetic behavior determines the dimensionality of a ferromagnetic material. Under some conditions, ferroelectric materials can only exist as a single domain. Fine particle magnetism originates from size effects, which are rooted in the magnetic domain structure of ferromagnetic materials. According to magnetic domain theory, the critical domain size is affected by a number of factors, such as the magnitude of magnetic saturation, the strength of crystal anisotropy and exchange forces, surface or domain-wall energy, and particle shape. Ferromagnetic materials' responses to applied fields can be understood by looking at two important properties: remanence and coercivity. The second one is related to the 'thickness' of the curve. Coercivity, a property that depends heavily on particle size, is one of the most crucial factors to think about when dealing with tiny particles. It has been demonstrated that the coercivity reaches a maximum and then falls to zero as the particle size is reduced.

1.4 Characteristics of Magnetic Nanoparticles

Magnetic anisotropy and shape are two of several characteristics that define the behavior of magnetic particles; size is one of them. Furthermore, Bloch barriers dissect the so-called magnetic domains. When an external magnetic field is not applied to a homogeneously magnetized particle, domain formation becomes more efficient due to the reduction of the particle's magnetic stray field. Domain formation is the root cause of this problem. No external magnetic field can ever magnetize a particle because its magnetization directions are statistically aligned in all domains. This describes the particle's magnetic moments in detail. Decreases in magnetic particle size are associated with an increase in the wall energy/total particle energy ratio. A whole particle displays unidirectional spontaneous magnetization when the size of the particle is so small that energy limitations prohibit the creation of magnetic domains. The orientation of a particle's magnetization is dictated by its crystal lattice, which is also known as the "easy axis." One property of single domain particles that determines their size is their form factor, which is defined as the ratio of their length in different directions to the magnetic field.

2. LITERATURE REVIEW

Rani, S. S. S., Radhika, S., & Padma, C. M. (2024) The co-precipitation procedure is employed to synthesize single-domain M-type strontium ferrite nanoparticles. Utilizing the XRD pattern, we determined that the crystallite size of the M-SrFe nanoparticles is 54.1%. The scanning electron micrographs reveal this hexagonal structure. An instance of M-SrFe nanoparticles is seen in the EDS examination. The sample is considered a hard magnetic material with elevated coercivity, as established by the VSM characterization tests. Hexaferrite is commonly employed in permanent magnets and recording devices because of its remarkable magnetic characteristics. The application of the Kubelka-Munk plot to the UV-DRS reflectance data revealed a band gap energy of 1.95 electron volts. PL analysis indicates that the wavelength with the highest intensity peak is 629.9 nm. The remarkable thermal stability of M-SrFe nanoparticles is corroborated by TG-DTA analyses. The four-probe approach is employed to ascertain the sample's resistivity, measured at 0.312 Ωm.

Aidin Lak, Sabrina Disch, Philipp Bender (2021) The energy and healthcare industries are only two of many that could benefit greatly from iron oxide nanoparticles. So far, the main emphasis has been on producing nanoparticles with a single crystal structure and no flaws. Recent research, on the other hand, has shown that defect-rich nanoparticles outperform defect-free ones in magnetic particle imaging (MPI) and magnetic hyperthermia. This paper's overarching goal is to provide a concise summary of the current state of iron oxide nanoparticle design and characterization with regard to defects and spin disorder. Cancer treatment and medicine delivery using magnetic nanoparticles take advantage of flaws and disorder when used in intracellular magnetic hyperthermia.

Mohamed S. A. Darwish, Hohyeon Kim, Hwangjae Lee, Chiseon Ryu, Jae Young Lee, and Jungwon Yoon (2019) Magnetic nanoparticles (MNPs) that have a high specific loss power and a minimal metal content are much sought after for use in hyperthermia applications. The co-precipitation procedure has been the most reliable method for producing magnetic nanoparticles. This has been the case throughout scientific history. The effectiveness of their hyperthermia approaches is frequently inadequate, which is widely regarded as the most significant obstacle to the development of effective cancer therapy options. Despite this, the hyperthermia effectiveness of ferrite magnetic nanoparticles has to be improved further due to the unique qualities that they possess. These properties include a significant magneto crystalline anisotropy and an increased coercivity.

Evgeny A. Gorbachev, Pavel E.Kazin (2018) The magnetic composite SrFe10.7Al1.3O19/Co was manufactured by first reducing cobalt ions on the surface of hexaferrite particles in a solution of ethylene glycol, and then drying the particles after the reduction process was completed. A substance that was composed of submicron-sized hexaferrite particles that exhibited magnetic hardness and were covered with soft magnetic cobalt nanoparticles was utilized in the production of a composite material. Additionally, hexaferrite particles were utilized in the construction of the substance. Within the context of this experiment, the utilization of the composite material made it possible to demonstrate the exchange-coupling effect that occurs between hard and soft magnetic processes.

Darja Lisjak, Petra Jenuš, and Alenka Mertelj (2014) The impact of morphology on the magnetic field configuration of ferrite nanoparticles was analyzed. Thin BaFe12O19 nanoplatelets were compared with hydrothermally produced isotropic, spherical, or octahedral CoFe2O4 nanoparticles. Employing "drop deposition," nanoplatelets and nanoparticles produced various hierarchical structures from stable liquids. In situ optical microscopy revealed the alignment of the nanoparticles within the magnetic field. Transmission and scanning electron microscopes were utilized to examine the morphologies of the nanoparticles and their ultimate assemblies. A vibrating-sample magnetometer was utilized to assess the magnetic characteristics of the nanoparticles and their aggregates. The BaFe12O19 nanoplatelets formed organized sheets several micrometers thick, exhibiting a magnetic alignment of around 90% in the substrate plane.

Subhasis Rana, John Philip & Baldev Raj (2010) Utilizing the reverse micelle technique, we successfully synthesized cobalt ferrite nanoparticles averaging 4 nm in size with a narrow size dispersion. The thermal history of samples was demonstrated to influence the distribution of cations and the phase transition of the samples. The FT-IR measurements indicated that cations were transitioning between octahedral and tetrahedral sites within the inverse spinel structure of CoFe2O4 nanoparticles, corroborating prior research. During heat treatment, the peak intensity of the tetrahedral sites increased at the expense of the octahedral sites, signifying that cations migrated from the octahedral to the tetrahedral sites due to this transition. The TGA/DTA investigation also confirmed the isochemical alteration that occurred throughout the heat treatment process.

3. MATERIALS AND METHODS

Magnetic nanoparticles are employed extensively in the field of biomagnetics for a wide range of applications, such as drug administration and sensing. Highly reactive ferrrite nanoparticles with precisely controllable composition, microstructure, size, and properties can be prepared using nontraditional bottom-up nanotechnology approaches, particularly wet chemical routes. These nanoparticles can then be tailored to meet the unique demands of a wide range of cutting-edge applications.

3.1 Magnetic and Mössbauer Characterization

For the purpose of collecting magnetic data, ferrite nanoparticle powders were immersed in a magnetometer inside a gelatin capsule that had been previously sealed in paraffin wax. The capsules were then placed into a plastic straw. Magnetization measurements were then taken with a Quantum Design Magnetic Property Measurement System (MPMS) at fields of up to 5 T and temperatures between 2 and 300 K. At room temperature (RT), samples were dispersed in boron nitride powder and studied using Mössbauer spectroscopy in their original form.

3.2 Methodology

The synthesis of MFe2O4 nanoparticles was carried out using a hydrothermal method, which was appropriately adjusted from an earlier study by Wang et al. that had concentrated on the production of MnFe2O4 nanorods. In particular, 16 mL of water was mixed with a 0.5 M solution of the metal precursor (e.g., CoCl2, NiSO4, ZnF2, Cu (NO3)2·3H2O, MgB2, FeCl2·4H2O, or FeCl3, depending on the desired ferrite composition) using magnetic stirring in a beaker with a micro (flea) type stir bar operating at approximately 300 rpm.

The solution was then adjusted to a concentration of 0.1 M by adding FeCl3. To ensure that the autoclave was filled to 80% capacity, the molarities of the precursor solutions was determined by considering the volume of the final solution, which was 18.3 mL. And lastly, with strong magnetic swirling, 2.3 mL of a NaOH solution with a concentration of 4.4 M was added to the beaker. Depending on the metal precursor used, a noticeable shift in color from orange/yellow to a dark brown/black tint occurred after these steps, indicating that the reaction was progressing. After 30 minutes of stirring with a micro (flea) type stir bar set at approximately 300 rpm, the solution was moved to a 23 mL Teflon-lined stainless-steel autoclave (by Parr Instruments) and sealed. The autoclave was typically heated to 190°C for 3–12 hours during a standard synthesis. Using surfactants like NP9 and APTES at the reaction temperatures indicated earlier allowed us to control the size regime of the ferrites. The autoclave was preheated to 220°C for 12 hours, specifically for crystalline ultra-small ferrites, and subsequently left to cool down to ambient temperature naturally. Following this, the product was rinsed four times with portions of distilled water and once with ethanol. It was then separated using centrifugation and decantation. To remove any remaining solvent, it was dried in a drying oven set at around 80°C. The final product was around 150 mg of sample.

3.3 Methods for Preparing Hexaferrites

Strontium hexaferrite has been manufactured by the application of a variety of manufacturing processes. The solid-state method, the salt melting method, ball milling, chemical co-precipitation, microemulsion, and self-propagating high temperature synthesis are some of the methods that involve these processes. Ferrites that are exceedingly pure and chemically homogeneous, with a size of nanometers, are produced by the micro emulsion pathway, which is an alternative and encouraging approach to the chemical synthesis methodology.

Three different chemical processes are utilized in this investigation in order to produce nanoparticles of SrFe12O19 hexaferrite. Self-propagating high temperature synthesis (SHS), chemical co-precipitation, and microemulsion are the three approaches that are utilized. The alternative procedures, which include the ceramic route and the sol gel process, amongst others, have a number of limitations, including the presence of impurities, the large grain size of the completed product, the requirement for high temperature sintering, and other similar issues.

4. RESULTS AND DISCUSSIONS

In the field of materials science, magnetic nanoparticles (MNPs) find use in numerous different contexts. Understanding the atomic structure, surface features, and magnetic properties of MNPs is crucial for designing them for specific applications. However, the success of these designs in different fields is dependent on how stable the particles are under different conditions.

The ability to precisely adjust the magnetic characteristics of CoFe204 by adding or removing divalent cations is one of the material's main selling points. Unfortunately, the produced nanoparticles often had a highly aggregated and irregular shape, rendering them unusable for many applications. Development of protective measures to chemically stabilize bare MNPs against degradation during or after synthesis by coating them with organic or inorganic substances is thus vital for various applications.

4.1 Size, Composition, and Morphology of As-Generated Ferrites

During the process of developing our ferrite system "family," we tried out a large number of transition metal precursors in a systematic manner under standard synthetic conditions (i.e., 190 degrees Celsius for twelve hours without surfactant). By adhering to these protocols, we were able to produce ferrite nanoparticles that had a variety of different chemical compositions. To provide a more explicit explanation, we were able to generate nanoparticles of MgFe2O4, CoFe2O4, NiFe2O4, CuFe2O4, and ZnFe2O4 by employing precursors of MgB2, CoCl2, NiSO4 (or Ni(acac)2), Cu (NO3)2·3H2O, and ZnF2, respectively. On the other hand, even though their effectiveness varied, other precursors were also effective.

4.2 Experimentation with Magnetic Properties

The electron spine resonance (ESR) examination is an essential part of the process of analyzing the magnetic properties of ferrite materials. Magnetic resonance in ferrite is caused by the interaction of spin with electromagnetic waves from ESR. This interaction is what creates the magnetic resonance. Ferrites have the most powerful A-B site interaction of all the material types. Both NiCr0.07Fe1.93O4 and NiCo0.07Fe1.93O4 ferrites have powder ESR spectra that are which was taken at ambient temperature. In addition, the g-factor of the electron was calculated using. The patterns that are displayed by both ferrites are identical, as can be seen in the photos. This occurs as a result of the presence of unpaired electrons within the compounds, which causes them to exhibit signals that are both broad and symmetrical. Based on this, it can be concluded that NiCr0.07Fe1.93O4 and NiCo0.07Fe1.93O4 both possess a property known as paramagnetic characteristics. The resonance magnetic field of NiCr0.07Fe1.93O4 ferrite is 2.2516 at 300 Gauss, which is lower than that of NiCo0.07Fe1.93O4 ferrite, which is 2.2514 at 300 Gauss. This is something that can be observed based on the comparison of the two samples. According to the findings of the X-ray diffraction analysis, the crystallite size of NiCr0.07Fe1.93O4 is larger than that of NiCo0.07Fe1.93O4. As a result, the magnetic interaction between the NiCr0.07Fe1.93O4 ferrite and the surrounding environment will be enhanced.

4.3 Study of Nanoparticle Materials

The study of nanoparticles has grown in prominence as of late within the realm of materials science. Physical properties of nanoscale particles (10 to 100 nm) typically differ from those of big particles (10-100 µm) or species at the molecular or atomic level. Depending on their size, nanoparticles display a wide range of physical characteristics, including surface reactivity, magnetic, optical, and other attributes that were previously undiscovered. Consolidated nano-materials exhibit enhanced mechanical capabilities, as demonstrated in recent investigations. These qualities include enhanced hardness of metals and enhanced ductility and plasticity of ceramics.

The electrical, optical, magnetic, and chemical characteristics of these nanoparticluate materials are frequently far more intriguing than those of their bulk equivalents. For multi-terabit magnetic storage devices, the production of discrete magnetic nanoparticles, with diameters varying from 10 to 100 nm, is crucial. Ferrofluids, magnetic refrigeration systems, MRI contrast enhancement, drug targeting, and catalysis are some of the potential uses for magnetic nanoparticles. These nanoparticles dissolve in a variety of organic solvents, and then their powdered form is obtained simply by rinsing off the solvent.

5. CONCLUSION

Ni-Zn ferrite nanoparticles are extremely attractive for application in electronic, biological, and electrical devices due to the excellent features that they possess. A few examples of these characteristics are low coercivity, low magnetic losses, high Curie temperature, high resistivity, mild saturation magnetization, great mechanical hardness, chemical stability, high magnetic permeability, and resistance to corrosion. Within the scope of this study, we were able to enhance the magnetic properties of the Ni-Zn ferrite system by modifying the annealing temperature, the synthesis techniques, and the doping concentration. Copper ions have been utilized as dopants due to the fact that they possess a high curie temperature, a positive magneto crystalline anisotropy energy, and a powerful magnetic moment.

Burning hexaferrite at moderate temperatures is the most effective way to preserve the structural integrity and perfection of the material. It is also possible that the possibility of discontinuous grain growth occurring will reduce when the sintering temperature is lowered. When it comes to determining the particle size of the final product, the techniques that are employed for powder synthesis are crucial. Annealing methods allow for the refinement of the crystal structure and the removal of imperfections that have an effect on the magnetic properties of the material.

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