A PROJECT ON

“Synthesis and Characterization of RFeO3 (R=Ce, La, Y) nanoparticle”

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BY,
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M.Sc. Physics (IV Semester)
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UNDER THE SUPERVISION OF,

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CERTIFICATE BY SUPERVISOR

This is to certify that, **Nahid Fatima**; a student of M. Sc. (Physics-IV Semester) has successfully completed the dissertation on **"Synthesis and Characterization of RFeO3 (R=Ce, La, Y) nanoparticles"** under my supervision during the year 2022-2023. It is certified that the work has not been submitted anywhere else for the award of any other diploma or degree of this or any other University.

I wish her good luck and bright future.

Date:10 June 2021
Place: Lucknow

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CERTIFICATE BY HOD

This is to certify that **Nahid Fatima** is a student of M. Sc. (Physics-IV Semester) Session 2021-22 to 2022-23 at Integral University, Lucknow. She completed her project entitled **"Synthesis and Characterization of RFeO3 (R=Ce, La, Y) nanoparticle"** successfully under the supervision of Ms. Tahira Khatoon, Assistant Professor (Level II), Department of Physics, Integral University.

I wish her good luck and bright future.

Srivastava

Date:10 June 2023
Place: Lucknow

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Nahid Fatima

NAHID FATIMA

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List of Abbreviations

YFeO3	Yttrium Ortho Ferrite
RFeO3	Rare Earth Ortho Ferrite
LaFeO3	Lanthanum Ortho Ferrite
CeFeO3	Cerium Ortho Ferrite
XRD	X-Ray Diffraction
UV-DRS	UV-Visible Diffused reflectance Spectroscopy
FTIR	Fourier Transform Infrared Spectroscopy
VOCs	Volatile Organic Compound
SOFCs	Solid oxide fuel cells
EDAX	Energy Dispersive X-Ray Analysis

Abstract

The dissertation explores the synthesis and characterization of $RFeO_3$ (R = Ce, La, Y) nanoparticles, focusing on their structural, magnetic, and optical properties. The study aims to investigate the potential applications of these nanoparticles in various fields such as catalysis, sensing, and biomedical applications. The research begins with a detailed review of the current literature on the synthesis methods and characterization techniques employed for $RFeO_3$ nanoparticles. Various synthesis routes, including sol-gel, hydrothermal, and solid-state methods, are evaluated, considering their advantages and limitations. Furthermore, characterization techniques such as X-ray diffraction (XRD) were utilized for structural properties of the synthesized nanoparticles. In the experimental section, $RFeO_3$ (R = Ce, La, Y) nanoparticles are synthesized using sol-gel auto combustion method. The structural characterization is carried out using XRD to determine the crystalline phases and lattice parameters of the synthesized nanoparticles. The optical properties of the synthesized $RFeO_3$ nanoparticles are investigated using UV-DRS. The results demonstrate the successful synthesis of $RFeO_3$ (R = Ce, La, Y) nanoparticles with controlled morphological and structural properties. The characterization data reveal the crystal structure, optical and vibrational properties of the nanoparticles. The dissertation contributes to the understanding of the synthesis-structure-property relationships of $RFeO_3$ nanoparticles and highlights their potential applications in fields such as catalysis, sensing, and biomedical science.

Keywords: $RFeO_3$ nanoparticles, synthesis, characterization, structural properties, optical properties.

1.INTRODUCTION

1.1.Overview of Nanomaterials

Nanomaterials have emerged as a transformative field of research with significant implications across various scientific disciplines and industries. At the nanoscale, materials exhibit unique properties and behaviours that differ from their bulk counterparts, making them highly attractive for numerous applications.

One of the defining characteristics of nanomaterials is their nanoscale dimensions, typically ranging from 1 to 100 nanometres. At this scale, materials undergo quantum confinement effects, where the behaviour of electrons and other particles becomes governed by quantum mechanics. This confinement leads to altered physical, chemical, and optical properties compared to the same material in bulk form. For example, nanomaterials often exhibit enhanced strength, increased surface area, and modified optical properties, which can be exploited for various applications.

1.2.Significance of Nanomaterials

The emergence of nanomaterials has been driven by the need for advanced materials with enhanced performance and functionality. The ability to control and manipulate materials at the nanoscale has paved the way for the development of innovative technologies with superior efficiency, sensitivity, and durability. As a result, there has been an increasing focus on the synthesis, characterization, and utilization of nanomaterials to harness their immense potential for addressing pressing societal challenges (1).

The significance of nanomaterials lies in their potential to revolutionize fields such as electronics, medicine, energy, catalysis, and environmental science. In electronics, nanomaterials enable the development of miniaturized devices with improved performance, such as faster and more efficient transistors or smaller, higher-capacity memory storage. In medicine, nanomaterials can be utilized for targeted drug delivery, imaging, and biosensing, offering improved diagnostics and therapies. In the energy sector, nanomaterials are being explored for efficient solar cells, energy storage devices, and catalysts for sustainable energy conversion processes. Moreover, nanomaterials find applications in environmental science, enabling advanced pollution remediation, water purification, and environmental monitoring (2).

1.3.Perovskite Materials

Perovskite materials belong to a specific class of compounds that exhibit a distinctive crystal structure known as the perovskite structure. This crystal structure is characterized by an arrangement of cations and anions in a specific geometric pattern, forming a three-dimensional framework. The general chemical formula of perovskites is ABX_3, where A and B are cations, and X represents an anion.

1.4.Crystal Structure of Perovskites

The crystal structure of perovskite materials is characterized by a three-dimensional framework of corner-sharing octahedra. In this structure, the A cation occupies the corners of the unit cell, the B cation is situated at the center of each octahedron, and the X anion forms a face-centered cubic arrangement around the octahedra. This arrangement creates a stable and symmetrical crystal lattice.

The flexibility of perovskite materials stems from the ability to substitute different cations and anions within the crystal structure. The A-site cation can be chosen from a wide range of elements, including alkaline earth metals, rare earth metals, and transition metals. The B-site cation can also vary, allowing for additional compositional flexibility. Furthermore, the X anion can be chosen from elements such as oxygen, fluorine, or chlorine. This versatility in composition enables tailoring of the physical, chemical, and electronic properties of perovskite materials.

The specific arrangement of atoms in the perovskite crystal structure leads to a range of intriguing properties. For instance, some perovskite materials exhibit high dielectric constants, making them useful for capacitors and electronic devices. Others display ferroelectric properties, meaning they exhibit spontaneous electric polarization that can be reversed by applying an external electric field. This ferroelectric behavior is valuable for memory storage and piezoelectric applications (3).

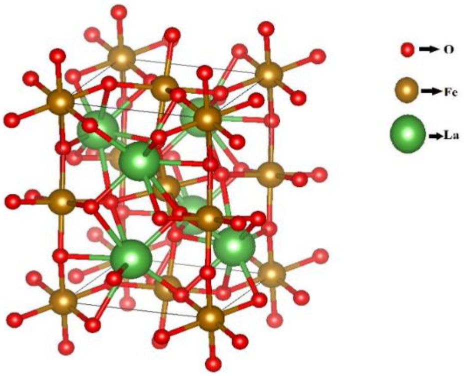

Figure 1 Crystal Structure of $RFeO_3$

Additionally, perovskite materials have shown superconductivity at low temperatures, making them attractive for applications in quantum computing and energy transmission. Some perovskites exhibit colossal magnetoresistance, a phenomenon where the electrical resistance changes dramatically in the presence of a magnetic field, which is important for magnetic sensors and data storage devices. Moreover, perovskites have been studied for their catalytic activity in various chemical reactions, such as oxygen reduction and water splitting, leading to applications in fuel cells and clean energy production.

1.5.Properties of Perovskite Materials

Perovskite materials exhibit diverse physical and chemical properties, making them highly versatile for various applications. These properties include high dielectric constants, ferroelectricity, superconductivity, magnetoresistance, photovoltaic activity, and catalytic behavior. The tunability of these properties by modifying the composition and structure of perovskite materials makes them highly desirable for technological advancements.

1.5.1. Optical Properties

Perovskite materials exhibit exceptional optical properties that have attracted significant attention for various applications. Many perovskites are direct bandgap semiconductors, meaning they efficiently absorb and emit light across a wide range of wavelengths. This property makes them highly suitable for optoelectronic devices such as solar cells, light-emitting diodes (LEDs), and photodetectors.

The tunability of the bandgap in perovskite materials is a particularly advantageous characteristic. By modifying the composition and structure, the bandgap of perovskites can be precisely engineered to match specific energy levels, allowing efficient absorption of photons in the visible and near-infrared regions. This tunability enables the design of perovskite solar cells with high power conversion efficiencies, rivalling traditional silicon-based solar cells.

Perovskite LEDs have also shown impressive performance, emitting bright and vibrant colors across the visible spectrum. The ability to precisely control the bandgap and charge transport properties of perovskites has led to the development of efficient and versatile light-emitting materials for display technologies and solid-state lighting (4).

Furthermore, perovskite materials have demonstrated exceptional photophysical properties, including high photoluminescence quantum yields, long carrier lifetimes, and low trap densities. These characteristics contribute to efficient light emission and charge transport, making perovskites promising candidates for next-generation optoelectronic devices.

1.5.2. Thermal Properties

The thermal properties of perovskite materials play a crucial role in determining their behaviour and suitability for different applications. Thermal conductivity refers to the ability of a material to conduct heat. Perovskite materials generally exhibit moderate to high thermal conductivities, which make them effective heat dissipators. This property is important in applications where efficient heat transfer is required, such as in thermal management of electronic devices and heat exchangers. The thermal expansion coefficient of a material describes how its dimensions change with temperature variations. Perovskite materials typically exhibit moderate thermal expansion coefficients, indicating that their crystal lattice expands or contracts to some extent with increasing or decreasing temperature (5). This property can be critical in engineering applications where dimensional stability is important, as excessive thermal expansion or contraction can lead to mechanical stress and material failure. Thermal stability is a key characteristic of perovskite materials, particularly for applications that involve high-temperature environments. Perovskites are known for their ability to maintain their structural integrity and desired properties over a wide temperature range. This thermal stability allows perovskite materials to withstand harsh operating conditions without significant degradation or phase transformations.

1.5.3. Electronic Properties

The electronic properties of perovskite materials play a vital role in their applications in electronics and energy conversion devices. Perovskites can exhibit a range of electrical conductivity behaviours, including semiconducting, metallic, and superconducting properties.

In the case of semiconducting perovskites, their bandgap determines their ability to conduct electricity. By engineering the bandgap through composition and structural modifications, perovskites can be tailored for specific electronic applications. This tunability has enabled the development of high-performance transistors, sensors, and memory devices.

Some perovskite materials also display metallic behaviour, characterized by high electrical conductivity and the absence of an energy bandgap. This property makes them suitable for applications such as interconnects, electrodes, and transparent conductive coatings (6).

Furthermore, certain perovskite compounds exhibit superconductivity at low temperatures, where they can conduct electric current without resistance. Superconducting perovskites have implications for the advancement of energy transmission and storage, as well as quantum computing technologies.

1.5.4. Magnetic Properties

Perovskite materials can exhibit intriguing magnetic properties that make them relevant for applications in data storage, sensing, and spintronics. Some perovskites display ferromagnetism, where neighbouring magnetic moments align parallel to each other, resulting in a net magnetic field. This behaviour is crucial for the development of magnetic memories and magnetic sensors (7).

Additionally, certain perovskites exhibit antiferromagnetic properties, where adjacent magnetic moments align in an alternating fashion, resulting in a net magnetic moment of zero. Antiferromagnetic perovskites have shown potential for spintronics applications, where the manipulation of electron spins enables the development of more efficient and power-saving electronic devices (8).

1.6.Synthesis Methods

Several synthesis methods have been employed for the preparation of perovskite materials, including sol-gel, hydrothermal, solid-state reaction, chemical vapor deposition, and template-assisted methods. Each method offers distinct advantages in terms of control over composition, morphology, and crystallinity of the resulting perovskite materials.

1.6.1. Solid-State Reaction Method

The solid-state reaction method involves the direct mixing and heating of precursor materials to induce a chemical reaction and the formation of the desired perovskite phase. This method is relatively simple and cost-effective, making it suitable for large-scale production of perovskite materials.

1.6.2. Hydrothermal Method

The hydrothermal synthesis method involves the reaction of precursors in a high-pressure, high-temperature aqueous environment. This technique enables the synthesis of high-quality perovskite materials with well-defined crystal structures, narrow particle size distributions, and enhanced purity.

1.6.3. Sol-Gel Method

The sol-gel method involves the formation of a colloidal solution (sol) that undergoes gelation to form a solid network (gel), which is subsequently annealed to obtain the desired perovskite phase. This technique allows precise control over the chemical composition, homogeneity, and purity of the perovskite materials.

1.6.4. Sol-Gel Auto-Combustion Method

The sol-gel auto-combustion method is a popular and versatile technique for the synthesis of perovskite materials. This method involves the formation of a precursor solution, known as a sol, which undergoes self-sustained combustion to yield the desired perovskite product.

The synthesis process begins by dissolving metal salts or complexes in a suitable solvent to form a sol. The sol is typically a viscous liquid that contains metal ions in a dispersed state. To ensure the formation of a perovskite structure, appropriate stoichiometric ratios of the metal precursors are crucial.

After the sol formation, the auto-combustion step is initiated. This is achieved by adding a combustion agent, such as urea or glycine, to the sol. The combustion agent acts as a fuel source that undergoes rapid exothermic decomposition upon heating. The combustion reaction generates a high amount of heat, causing the sol to ignite and self-propagate.

The combustion reaction is usually self-sustaining, meaning it continues without the need for additional external heating. This feature makes the sol-gel auto-combustion method highly efficient and convenient.

1.7.Applications of Perovskites

1.7.1. Photovoltaic Applications

Perovskite materials have emerged as promising candidates for next-generation solar cells due to their exceptional light absorption properties, high carrier mobility, and low-cost processing. Perovskite solar cells have achieved remarkable efficiency improvements, rivalling the performance of traditional silicon-based solar cells (5).

1.7.2. Catalytic Applications

Perovskite materials exhibit excellent catalytic activity, making them suitable for applications such as oxygen reduction reactions, fuel cells, and pollution control. The unique electronic structure and surface properties of perovskite catalysts contribute to their high efficiency and selectivity in various catalytic processes (6).

1.7.3. Energy Storage Applications

Perovskite materials have shown potential for energy storage applications, including rechargeable batteries and supercapacitors. Their high energy density, long cycling stability, and rapid charge-discharge rates make them promising candidates for the next generation of energy storage devices (3).

1.7.4. Sensing applications

Perovskite materials have also demonstrated excellent sensing and detection capabilities for various gases, humidity, and chemical species. Their sensitivity, selectivity, and response speed make them suitable for applications in environmental monitoring, healthcare diagnostics, and industrial safety (4).

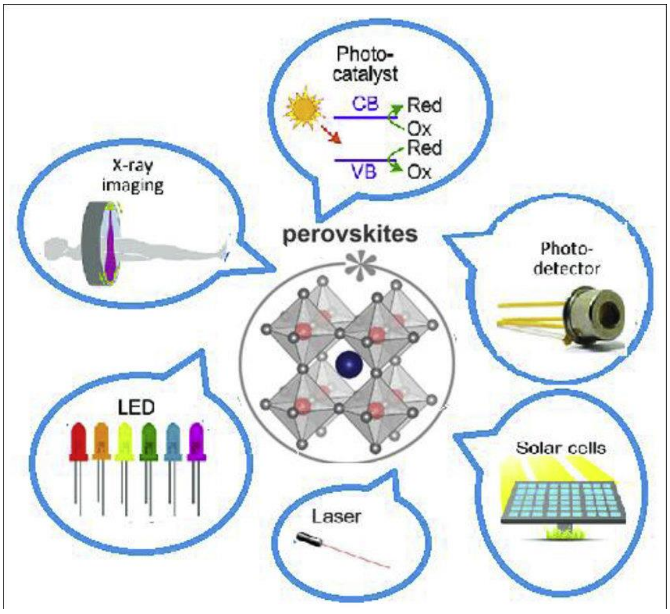

Figure 2 Applications of Perovskites

1.8.Objectives of Study

a) Synthesis of RFeO3 perovskite nanomaterials using Sol-gel Auto Combustion method, where R is rare earth element (R= Ce, La, Y).

b) Structural characterization of the synthesized materials using XRD.

c) Optical properties of synthesized materials using UV-DRS.

d) Vibrational properties using FTIR Spectroscopy.

2. LITERATURE REVIEW

$RFeO_3$ perovskite materials have shown exceptional catalytic activity in a range of reactions. For instance, $CeFeO_3$-based perovskites have been utilized as catalysts for the oxidation of volatile organic compounds (VOCs) due to their high surface area and redox properties. $LaFeO_3$-based perovskites have been employed as catalysts for the oxidation of methane, ethane, and other hydrocarbons. $YFeO_3$ perovskites have been investigated for the catalytic reduction of NO_x emissions from automobile exhaust. The unique structural and compositional properties of $RFeO_3$ perovskites contribute to their high catalytic efficiency and make them suitable for environmental remediation processes. $RFeO_3$ perovskite materials have shown great promise in the field of solid oxide fuel cells (SOFCs). These materials can be used as cathodes, anodes, or electrolytes in SOFCs. For example, $CeFeO_3$-based perovskites have been employed as cathode materials due to their high oxygen reduction activity and stability under operating conditions (9). $LaFeO_3$-based perovskites have demonstrated good electrochemical performance as anode materials in SOFCs. $YFeO_3$ perovskites have been investigated as electrolytes for SOFCs due to their high ionic conductivity. The incorporation of rare-earth elements (Re) in $RFeO_3$ perovskites enhances their performance and stability, making them promising candidates for advanced energy conversion devices. $RFeO_3$ perovskite materials have also shown potential for applications in magnetic devices. The doping of Re (Ce, La, Y) in $RFeO_3$ perovskites can lead to interesting magnetic behaviour, such as ferromagnetism or antiferromagnetic, depending on the specific composition and doping levels. For instance, $CeFeO_3$ perovskites have exhibited ferromagnetic behaviour, making them suitable for spintronic applications. $LaFeO_3$-based perovskites have been investigated for their magnetoresistance properties. $YFeO_3$ perovskites have shown multiferroic behaviour, with simultaneous magnetic and ferroelectric properties, opening possibilities for new multifunctional devices. The unique magnetic properties of $RFeO_3$ perovskites make them attractive for magnetic sensors, data storage systems, and other magnetic devices. $RFeO_3$ perovskite materials have shown potential in various electrochemical devices, including rechargeable batteries, supercapacitors, and gas sensors. The high ionic conductivity and tailored compositions of $RFeO_3$ perovskites enable efficient charge transport and electrochemical reactions (10). For example, $CeFeO_3$ perovskites have been investigated as electrode materials in lithium-ion batteries due to their high capacity and cycling stability. $LaFeO_3$-based perovskites have shown promise as supercapacitor electrodes. $YFeO_3$ perovskites have demonstrated gas-sensing properties, particularly for the detection of CO and NO_2 gases. The tuneable properties of $RFeO_3$ perovskites make them promising candidates for various electrochemical applications. $RFeO_3$ perovskite materials have recently gained attention for their photocatalytic properties. These materials can harness solar energy to drive photochemical reactions, such as water splitting, pollutant degradation, and CO_2 reduction. $CeFeO_3$-based perovskites, for instance, have been employed as photocatalysts for water splitting. $LaFeO_3$-based perovskites have shown promise in the degradation of organic pollutants under visible light irradiation (11). $YFeO_3$ perovskites have been investigated for CO_2 reduction to produce value-added chemicals. The unique band structure and electronic properties of $RFeO_3$ perovskites make them effective photocatalysts, holding potential for sustainable energy conversion and environmental applications. $RFeO_3$ perovskite materials have shown promise in gas sensing applications. Their unique surface properties, high sensitivity, and selectivity make them suitable for detecting various gases. $CeFeO_3$-based perovskites have been investigated for the detection of toxic gases such as carbon monoxide (CO) and nitrogen dioxide (NO_2). $LaFeO_3$ perovskites have demonstrated high sensitivity towards reducing gases such as hydrogen (H_2) and ammonia (NH_3). $YFeO_3$-based perovskites have been explored for the detection of hydrogen sulfide (H_2S) and other sulfur-containing gases. The development of $RFeO_3$ perovskite-based gas sensors holds great potential for environmental monitoring, industrial safety, and healthcare applications (12). $RFeO_3$ perovskite materials have also been investigated for heterogeneous photocatalysis applications. Their ability to absorb visible light and generate electron-hole pairs makes them suitable for various photocatalytic reactions. $CeFeO_3$ perovskites have been utilized for the degradation of organic pollutants, such as dyes and pharmaceuticals, in wastewater treatment. $LaFeO_3$-based perovskites have shown promise in the photocatalytic reduction of CO_2 to produce renewable fuels and chemicals. $YFeO_3$ perovskites have been explored for the degradation of organic pollutants and the removal of heavy metals from contaminated water (13). The development of $RFeO_3$ perovskite-based photocatalysts can contribute to the advancement of sustainable energy generation and environmental remediation. $RFeO_3$ perovskite materials have demonstrated potential in electrochemical water splitting, a key process for renewable hydrogen production. $CeFeO_3$-based perovskites have shown promise as efficient oxygen evolution catalysts in alkaline electrolytes. $LaFeO_3$ perovskites have been investigated as both anode and cathode materials for water splitting, demonstrating good electrocatalytic activity. $YFeO_3$-based perovskites have exhibited high activity as oxygen evolution catalysts under alkaline conditions. The utilization of $RFeO_3$ perovskites in electrochemical water splitting can contribute to the development of clean and sustainable hydrogen fuel production technologies. $RFeO_3$ perovskite materials have shown potential for gas separation applications and membrane technology. $CeFeO_3$-based perovskites have been explored for oxygen permeation membranes in oxygen separation processes, such as air separation and oxyfuel combustion. $LaFeO_3$ perovskites have demonstrated promise as proton-conducting membranes in hydrogen separation and purification. $YFeO_3$-based perovskites have shown potential in mixed ionic-electronic conducting membranes for oxygen permeation and hydrogen separation (6). The tuneable properties of $RFeO_3$ perovskites and their ability to exhibit high oxygen or proton conductivity make them attractive candidates for gas separation applications and the development of energy-efficient membrane technologies. $RFeO_3$ perovskite materials have been investigated for electrocatalytic applications, particularly in water splitting, fuel cells, and electrolysers. $CeFeO_3$-based perovskites have shown promise as electrocatalysts for the oxygen reduction reaction (ORR) in fuel cells. $LaFeO_3$ perovskites have been explored as bifunctional electrocatalysts for both the ORR and oxygen evolution reaction (OER) in rechargeable metal-air batteries. $YFeO_3$-based perovskites have demonstrated potential as efficient electrocatalysts for the hydrogen evolution reaction (HER) in water splitting. The development of $RFeO_3$ perovskite-based electrocatalysts can contribute to the advancement of clean energy technologies by improving the efficiency and performance of electrochemical devices. $RFeO_3$ perovskite materials have shown promise in thermoelectric applications, where they can convert waste heat into electrical energy (14). $CeFeO_3$-based perovskites have been investigated for their thermoelectric properties, demonstrating high Seebeck coefficients and good electrical conductivity. $LaFeO_3$ perovskites have shown potential as thermoelectric materials due to their low thermal conductivity and suitable electrical properties. $YFeO_3$-based perovskites have exhibited promising thermoelectric performance, attributed to their electronic band structure and crystal structure. The unique combination of properties in $RFeO_3$ perovskites makes them attractive candidates for the development of efficient thermoelectric materials for waste heat recovery and energy harvesting applications.

3. MATERIALS AND METHODS

3.1.Materials Used

The materials used in the preparation of $RFeO_3$ (R= Ce, La, Y) are Ferric Nitrate Nonahydrate ($Fe(NO)_3.9H_2O$), Cerium Nitrate Hexahydrate ($Ce(NO_3)_3.6H_2O$), Lanthanum Nitrate Hexahydrate ($La(NO_3)_3.6H_2O$), Yttrium Nitrate Hexahydrate ($Y(NO_3)_3.6H_2O$), Citric Acid Monohydrate ($C_6H_8O_7.H_2O$), Ethylene Glycol ($C_2H_6O_2$).

3.2.Synthesis of RFeO3

$RFeO_3$ (R= Ce, La, Y) fine particles were produced using the sol-gel auto-combustion method with citric acid as the fuel. Deionized water was used to dissolve metal nitrates and citric acid in stoichiometric amounts. To obtain brown gel, the mixture was progressively heated at 70 °C while constantly stirring for 180 minutes. The dried brown gel was then burned in a self-perpetuating combustion process until the gel had completely turned to powder. Finally, to achieve perfect crystallization, the produced orthoferrite powders were calcined at 800 °C for 4 hours. The resultants were collected and named Ce ($CeFeO_3$), La ($LaFeO_3$), and Y ($YFeO_3$) for further characterization.

3.3.Characterization Techniques

3.3.1. X-ray diffraction (XRD)

Figure 3 XRD Instrument

X-ray diffraction (XRD) is a powerful technique used to analyze the crystal structure of materials. It works on the principle of Bragg's law, which states that when X-rays strike a crystalline sample at a specific angle, they will be diffracted by the crystal lattice planes. The diffracted X-rays form a diffraction pattern, which can be detected and analyzed to determine the crystal structure and phase composition of the material. In the XRD setup, a beam of monochromatic X-rays is directed towards the sample at a specific angle, known as the incident angle or Bragg angle. The X-rays interact with the crystal lattice and are scattered by the atoms in the sample. The scattered X-rays interfere constructively when the path difference between them is equal to an integer multiple of the X-ray wavelength. This constructive interference leads to the formation of diffraction peaks in the XRD pattern. The XRD pattern is typically recorded by a detector that measures the intensity of the diffracted X-rays as a function of the scattering angle. By rotating the sample or the detector around the sample, a complete diffraction pattern can be obtained. The diffraction pattern consists of a series of peaks corresponding to different crystallographic planes in the material. The position and intensity of the diffraction peaks in the XRD pattern provide information about the crystal structure, lattice parameters, and phase composition of the material. The position of each peak corresponds to the spacing between crystal planes, while the intensity of the peak is related to the arrangement and population of atoms in the crystal lattice. The diffraction pattern can be analysed using mathematical techniques, such as the Rietveld method or peak fitting algorithms, to extract quantitative information about the crystal structure and phase composition.

3.3.2. UV-Visible Diffused Reflectance Spectroscopy

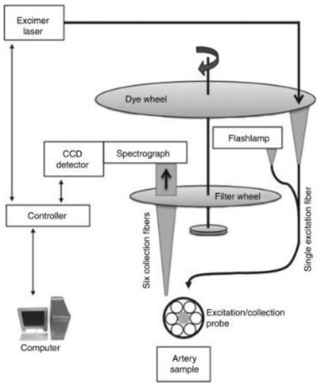

Figure 4 Schematic of DRS

UV-DRS (UV-visible diffuse reflectance spectroscopy) is a technique used to investigate the optical properties of materials, specifically their absorption and reflectance of light in the UV-visible range. The working principle of UV-DRS is based on the measurement of the diffuse reflectance of light from a sample. In UV-DRS, a UV-visible spectrophotometer equipped with an integrating sphere is used. The integrating sphere ensures that light is scattered evenly over the sample surface, allowing for accurate measurement of diffuse reflectance. The sample, typically in powdered form, is pressed into a pellet or coated onto a substrate and placed in the sample holder. In the UV-DRS setup, a broad spectrum of UV-visible light is directed towards the sample surface. The incident light interacts with the sample, and a portion of the light is absorbed by the material while the remaining light is reflected. The reflected light is collected by the integrating sphere, which measures the intensity of the diffuse reflectance as a function of wavelength. The collected diffuse reflectance data is typically converted into an absorbance or Kubelka-Munk function, which provides information about the optical properties of the material. The absorbance spectrum represents the amount of light absorbed by the sample at different wavelengths, revealing its absorption characteristics. The Kubelka-Munk function relates the diffuse reflectance to the absorption and scattering properties of the material, allowing for the determination of the bandgap energy. By analysing the UV-DRS data, important optical properties of the material can be obtained, including the bandgap energy, absorption edge, and optical band structure.

3.3.3. SEM and EDAX

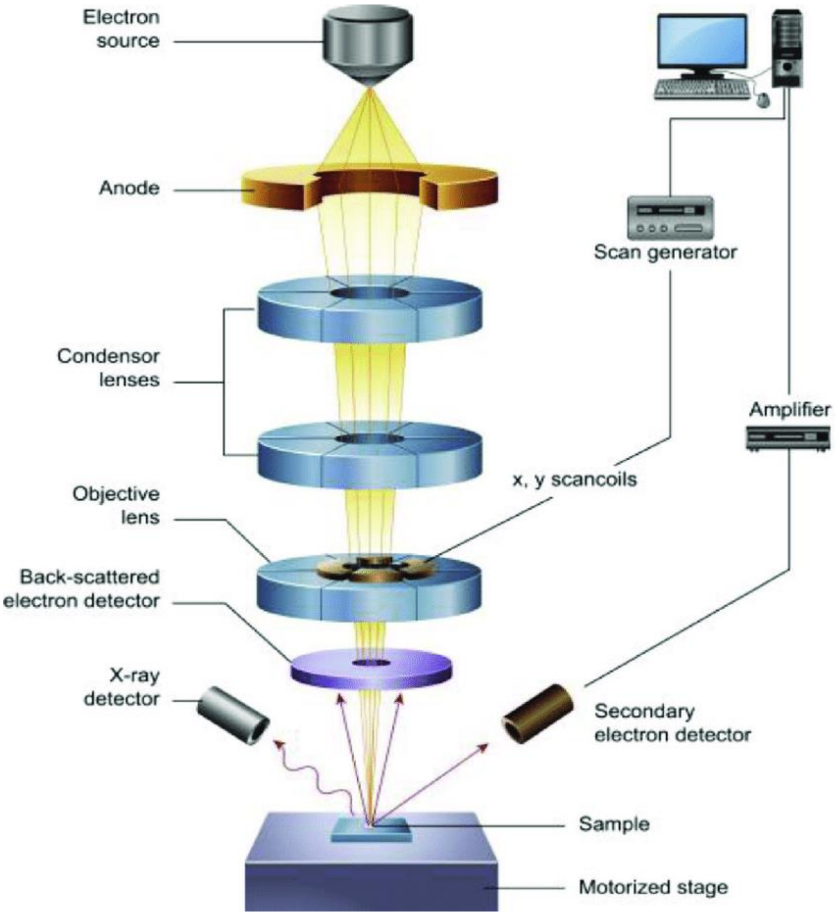

Figure 5 Schematic of SEM

Scanning Electron Microscopy (SEM) is an imaging technique used to examine the surface morphology, topography, and composition of materials at high resolution. It operates by scanning a focused beam of electrons across the sample surface and detecting various signals emitted from the interaction of the electrons with the sample. In combination with Energy Dispersive X-ray Analysis (EDX or EDAX), elemental composition analysis can also be performed. The working principle of SEM involves several key components. First, a beam of electrons is generated from an electron source, typically a heated filament or a field-emission source. These electrons are accelerated towards the sample by applying a high voltage. As the electrons interact with the sample, several signals are generated. One of the primary signals used in SEM imaging is secondary electrons (SE). These low-energy electrons are emitted from the sample surface due to the interaction of the primary electron beam with the atoms of the sample. The SEs are collected by a detector, and their intensity is used to generate the surface image, providing information about the sample's topography and morphology. In addition to secondary electrons, backscattered electrons (BSE) are also detected in SEM. BSEs are high-energy electrons that are deflected back towards the detector after interacting with the sample. The intensity of BSEs depends on the atomic number of the elements present in the sample, allowing for compositional contrast in the SEM image. EDX analysis is often integrated with SEM to provide information about the elemental composition of the sample. EDX works by detecting the characteristic X-rays emitted when high-energy electrons in the sample undergo transitions to lower energy states. The X-rays are collected by an energy-dispersive detector, and their energy is used to identify the elements present in the sample and determine their relative concentrations. By combining SEM imaging with EDX analysis, researchers can obtain detailed information about the surface morphology, topography, and elemental composition of the sample. This enables the characterization and identification of different phases, the analysis of particle size and distribution, and the investigation of elemental mapping and chemical analysis.

3.3.4. FTIR Spectroscopy

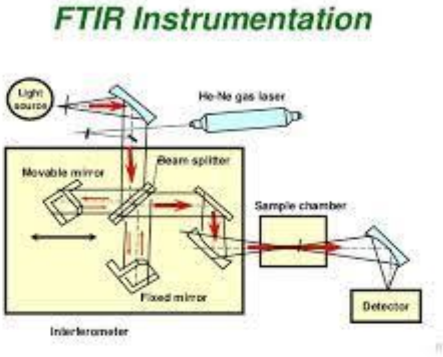

Figure 6 FTIR Spectrometer

Fourier-transform infrared spectroscopy (FTIR) is a widely used technique for analyzing the chemical composition and molecular structure of materials based on their interaction with infrared (IR) radiation. It works on the principle that molecules absorb specific frequencies of infrared light, resulting in characteristic vibrational modes that can be detected and analyzed. The working of FTIR spectroscopy involves several key components. First, a beam of IR radiation, typically generated by a broadband IR source, is directed towards the sample. The IR beam passes through an interferometer, which splits it into two beams: a sample beam and a reference beam. The sample beam passes through the sample, while the reference beam follows a separate path. The sample beam interacts with the sample, and the molecules in the sample absorb specific frequencies of the IR radiation, corresponding to their molecular vibrations. These absorptions result in a reduction in the intensity of the sample beam at specific wavelengths. The reference beam, on the other hand, does not interact with the sample and serves as a reference for comparison. After passing through the sample and the reference path, the two beams are recombined in the interferometer. The resulting combined beam contains interference patterns that are specific to the absorption characteristics of the sample. This combined beam is then directed towards a detector, which measures the intensity of the combined beam as a function of wavelength. The detector generates an interferogram, which is a plot of the intensity of the combined beam as a function of the optical path difference. This interferogram is then subjected to a mathematical technique called Fourier transformation to convert it into a spectrum representing the intensity of the absorbed IR radiation as a function of wavelength or wavenumber. The obtained FTIR spectrum provides valuable information about the functional groups, chemical bonds, and molecular structure of the sample. Each peak or band in the spectrum corresponds to a specific vibration or combination of vibrations within the molecules present in the sample. By comparing the observed absorption frequencies with reference spectra or databases, researchers can identify the functional groups and chemical bonds present in the sample.

4. RESULTS AND DISCUSSION

4.1.X-ray diffraction (XRD)

X-ray diffraction (XRD) analysis was performed to determine the crystalline structure and phase purity of the synthesized $RFeO_3$ (R= Ce, La, Y) perovskite nanomaterials. X-ray diffraction patterns were recorded using a high-resolution X-ray diffractometer. The XRD patterns were collected in the 2θ range of 10° to 80° using Cu Kα radiation (λ = 1.5406 Å). The obtained XRD plots are in accordance with previously reported literature (15,16). The peaks reveal Orthorhombic structure of $CeFeO_3$ and $LaFeO_3$, while a hexagonal structure of $YFeO_3$. WH plot was used to determine the average crystallite size. The summary of result obtained is listed in Table 1.

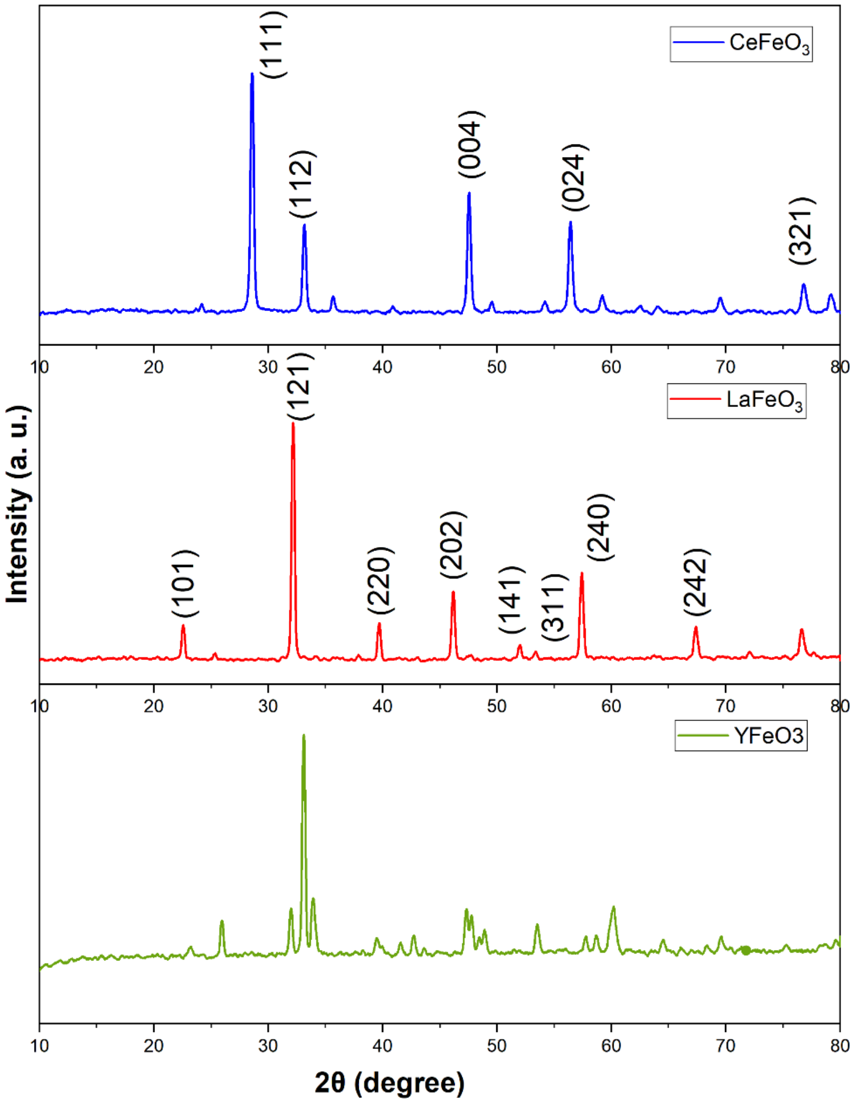

Figure 7 XRD of prepared samples

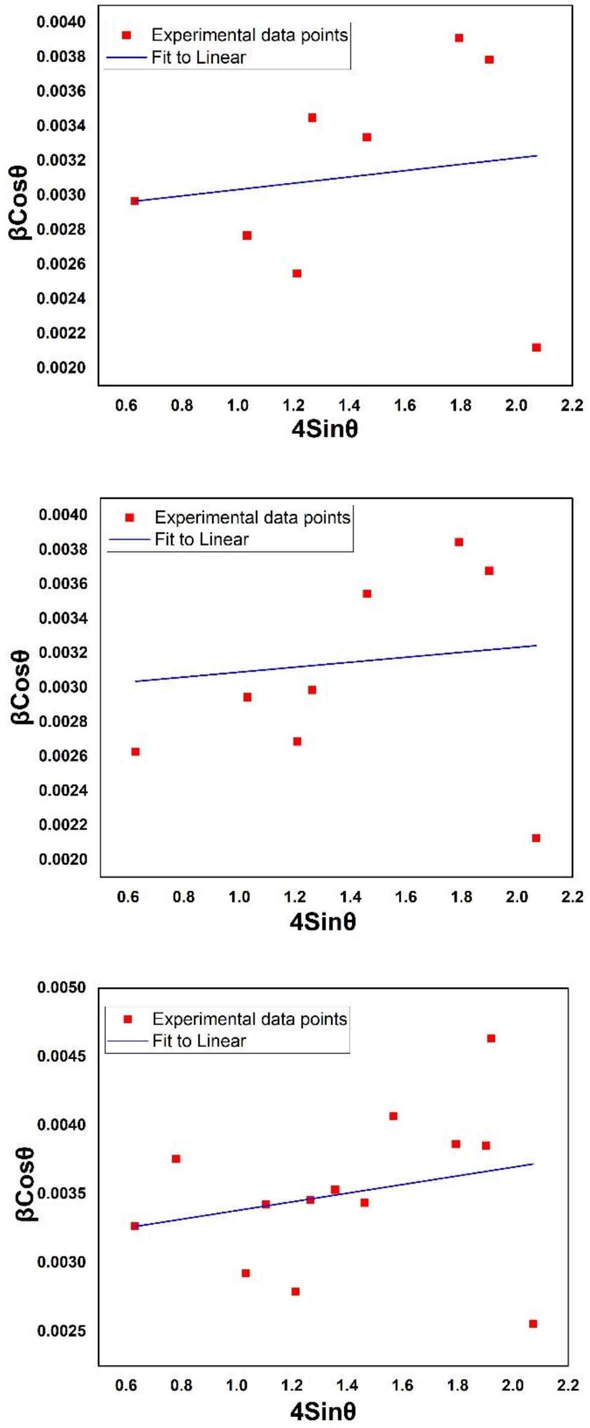

Figure 8 WH Plots

Sr. No.	Name of Sample	Crystallite Size (WH Plot)
1.	$CeFeO_3$	50.81
2.	$LaFeO_3$	49.09
3.	$YFeO_3$	41.61

4.2.UV-Visible Diffused Reflectance Spectroscopy

UV-DRS measurements were carried out to investigate the optical properties and bandgap energy of the $RFeO_3$ (R= Ce, La, Y) perovskite nanomaterials. The diffuse reflectance spectra were recorded using a UV-visible spectrophotometer equipped with DRS mode. The powdered samples were pressed into pellets and placed in the sample holder for analysis. The reflectance spectra were collected and Tauc plots were used to determine the band gap of prepared orthoferrites. Obtained band gaps after extrapolating Tauc plots of $CeFeO_3$, $LaFeO_3$ and $YFeO_3$ are 2.15 eV, 1.90 eV and 2.08 eV respectively.

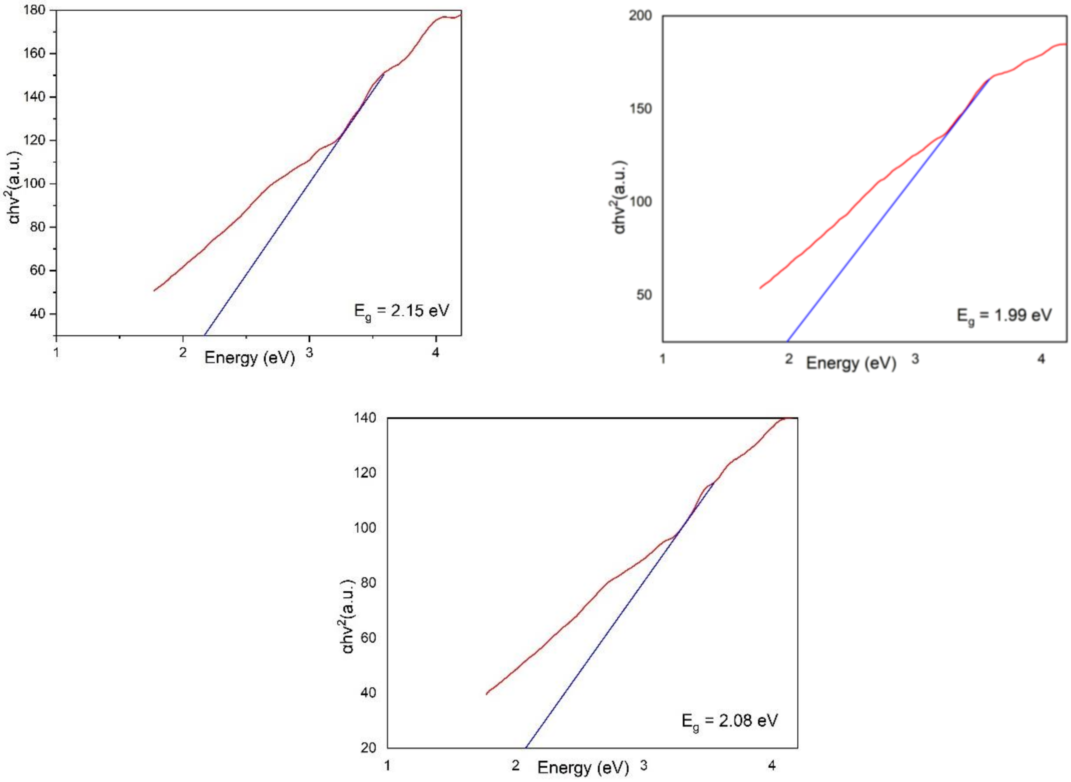

Figure 9 Tauc Plots

4.3.FTIR Spectroscopy

FTIR spectroscopy was employed to investigate the chemical bonding and functional groups present in the $RFeO_3$ (R= Ce, La, Y) perovskite nanomaterials. The powdered samples were mixed with an appropriate infrared transparent matrix (such as KBr) and compressed into pellets. The FTIR spectra were acquired using a Fourier-transform infrared spectrometer over a wavenumber range of 400-4000 cm^{-1}. The obtained FTIR spectra were analysed to identify the vibrational modes and characteristic peaks corresponding to the chemical bonds and functional groups in the perovskite nanomaterials.

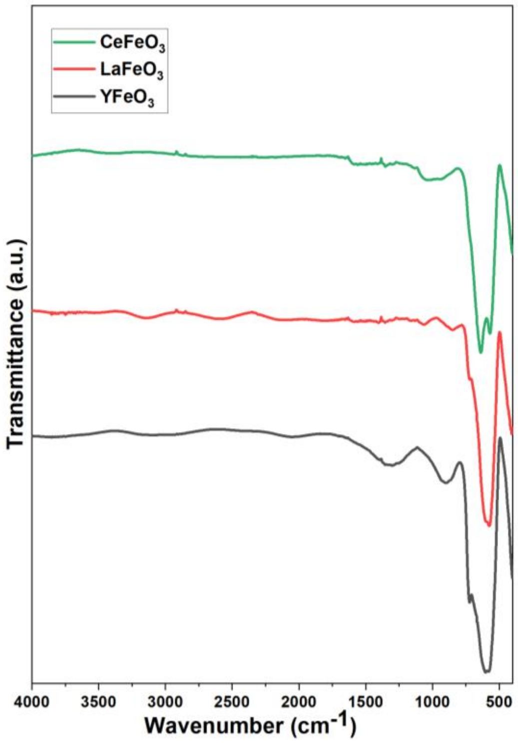

Figure 10 FTIR Spectra

5. CONCLUSION

In summary, the structural, and optical properties of the $RFeO_3$ (R= Ce, La, Y) perovskite nanomaterials were characterized using XRD, UV-DRS, EDAX and FTIR spectroscopy. These techniques provided valuable insights into the crystal structure, bandgap energy, morphology, chemical bonding, and functional groups of the perovskite nanomaterials, aiding in the understanding of their properties and potential applications. The results are in well agreement with previously reported research work. Prepared materials can be used further for several applications.

References:

1. Nanomaterials Handbook. Nanomater Handb [Internet]. 2006 Jan 26 [cited 2022 Mar 2]; Available from: https://www.taylorfrancis.com/books/mono/10.1201/9781420004014/nanomaterials-handbook-yury-gogotsi

2. Hasanzadeh M, Shadjou N, de la Guardia M. Iron and iron-oxide magnetic nanoparticles as signal-amplification elements in electrochemical biosensing. Vol. 72, TrAC - Trends in Analytical Chemistry. Elsevier B.V.; 2015. p. 1–9.

3. Barilo SN, Ges AP, Guretskii SA, Zhigunov DI, Ignatenko AA, Igumentsev AN, et al. Seeded growth of rare-earth orthoferrites from B2O3-BaF2-BaO solvent II. Growth of high-quality RFeO3 single crystals. J Cryst Growth. 1991 Jan 2;108(1–2):314–8.

4. Koshizuka N, Hayashi K. Temperature dependences of one-magnon light scattering in RFeO3. J Magn Magn Mater. 1983;31–34(PART 2):569–70.

5. Warshi MK, Mishra V, Sagdeo A, Mishra V, Kumar R, Sagdeo PR. Structural, optical and electronic properties of RFeO3. Ceram Int. 2018 May 1;44(7):8344–9.

6. Ameri I, Boularaf A, Drief F, Zaoui A, Kacimi S. Ab-initio study of magnetic and electronic properties of the perovskites RFeO3: 4f-R valence electrons effects. J Magn Magn Mater. 2021 Nov 1;537.

7. Gao C, Liu G, Liu X, Wang X, Liu M, Chen Y, et al. Flower-like n-Bi2O3/n-BiOCl heterojunction with excellent photocatalytic performance for visible light degradation of Bisphenol A and Methylene blue. J Alloys Compd. 2022 Dec 25;929:167296.

8. Noor M, Sharmin F, Mamun MAA, Hasan S, Hakim MA, Basith MA. Effect of Gd and Y co-doping in BiVO4 photocatalyst for enhanced degradation of methylene blue dye. J Alloys Compd. 2022 Feb 25;895:162639.

9. Akaba R. Growth spirals on RFeO3 single crystals. J Cryst Growth. 1974;24–25(C):537–40.

10. Li CL, Zheng SS, Barasa GO, Zhao YF, Wang L, Wang CL, et al. A comparative study on magnetic behaviors and magnetocaloric effect in heavy rare-earth antiferromagnetic orthoferrites RFeO3 (R = Dy, Ho and Er). Ceram Int. 2021 Dec 15;47(24):35160–9.

11. Rudowicz C. On magnetocrystalline anisotropy of Co2+ ions substituted in RFeO3 orthoferrites. Phys Lett A. 1974 Mar 11;47(2):169–70.

12. Mizoguchi Y, Onodera H, Yamauchi H, Kagawa M, Syono Y, Hirai T. Mössbauer spectra and magnetic susceptibilities of ultrafine hexagonal RFeO3 (R ≡ Eu, Yb) particles formed by the spray inductively coupled plasma technique. Mater Sci Eng A. 1996 Oct 30;217–218:164–6.

13. Abdellahi M, Abhari AS, Bahmanpour M. Preparation and characterization of orthoferrite PrFeO3 nanoceramic. Ceram Int. 2016;42(4):4637–41.

14. Datt G, Sen Bishwas M, Manivel Raja M, Abhyankar AC. Observation of magnetic anomalies in one-step solvothermally synthesized nickel-cobalt ferrite nanoparticles. Nanoscale. 2016;8(9):5200–13.

15. Nguyen TA, Pham V, Chau DH, Mittova VO, Mittova IY, Kopeychenko EI, et al. Effect of Ni substitution on phase transition, crystal structure and magnetic properties of nanostructured YFeO3 perovskite. J Mol Struct [Internet]. 2020;1215:128293. Available from: https://doi.org/10.1016/j.molstruc.2020.128293

16. Nakhaei M, Sanavi Khoshnoud D. Study on structural, magnetic and electrical properties of ReFeO3 (Re= La, Pr, Nd, Sm & Gd) orthoferrites. Phys B Condens Matter [Internet]. 2021;612(December 2020):412899. Available from: https://doi.org/10.1016/j.physb.2021.412899