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Analysis of Structural, Optical, Morphological and Magnetic Behaviour of Lead Nanoferrite by Two Different Methods

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Abstract
Sol gel and the hydrothermal approach are two distinct techniques that have been successfully used to synthesise lead nanoferrite ($PbFe_2O_4$). To determine which of the two approaches is superior, the features of the sample prepared by each have been examined. Vibration Sample Magnetometer (VSM), Fourier Transform Infrared Spectroscopy, UV-VIS Spectral investigations, Scanning Electron Microscopy (SEM), X-Ray Diffraction, and Size, Band Gap Energies, Morphology, and Magnetic Properties of Nanostructures were used to determine and compare its various aspects. The mean diameters of the synthesised lead ferrite nanocrystalline were found to be 33 nm for the sol gel method and 20 nm for the hydrothermal method, respectively, based on X-Ray Diffraction analysis using Debye-Scherer's formula with the peak. FTIR spectra are used to analyse the vibration characteristics of nanomaterials. The optical characteristics of the synthesised nanomaterials, such as their Energy Band Gap, were determined using UV-VIS Spectral investigations. SEM is used for surface morphological investigations, allowing the structure of the synthesised nanomaterials to be studied. Using VSM, the magnetisation of the produced nanomaterials was examined, and the hysteresis loops were used to calculate the saturation magnetisation (M_s), coercivity (H_c), and retainivity (M_r).
Keywords: Nanoferrites, Sol Gel Method, Hydrothermal Route, Comparative Method

Introduction

Larger specific area or smaller crystallite size are two ways that nanostructured materials differ from traditional bulk materials in that they display unique physical and chemical properties. That is why within the past ten years, there has been an increased focus on the creation and characterisation of nanomaterials. Because the nanostructured phase of the materials affects a variety of aspects in nanomaterials, including structural, surface reactivity, electrical, and magnetic properties (Murthy et al.; Shanmugam; Valenzuela).

Ferrites are a family of magnetic oxide ceramic materials that belong to the group of compounds mostly composed of iron oxide. Ferrites are a significant class of magnetic materials with a wide range of uses among other magnetic materials. Depending on the application, they encompass a wide range of frequencies, from radio frequency to microwave frequency, including electronic devices, Ferro fluids, magnetic medicine delivery, microwave devices, etc. Ferrites are utilized in a wide range of electronics, including catalysts, inductors, sensors, recording heads, magnetic cores, and information storage devices (Patange).

Their uses are spectacular and span from power handling, simple permanent magnets, magnetic recording, to millimetre wave integrated circuits. The foundation of these applications lies in ferrites' fundamental characteristics, which include strong electrical resistivity, low electrical losses, considerable saturation magnetisation, and excellent chemical stability (Sutka and Mezinskis; Goldman). Ferrites are obtainable in three distinct crystal systems by various techniques, and the ability to create an almost infinite number of solid solutions allows for the customisation of their characteristics for a wide range of uses. Ferromagnetic metals cannot replace ferrites in many applications, and ferrites frequently face competition from metals due to cost considerations (Glezer).

The ability to manufacture ferrites as nanoparticles has created a novel and fascinating area of study with innovative implications for both biotechnology and electronic technology.

Due to ferrites' tunable qualities, their use in daily life is growing. Because of their surface area and quantum confinement, nanoferrites are known to have better structural, electrical, magnetic, optical, and other properties than their bulk ferrite counterparts (Sugimoto).

Nanomaterial fabrication and applications are the subject of extensive research due to their distinct physical and chemical characteristics. Given the significance of these materials for both science and technology, they are highly appealing. The most important magnetic materials are nanocrystalline spinel ferrites, which have the common formula MFe_2O_4 (M = Pb, Ni, Zn, Mn, Co, Mg, etc.) (Verwey et al.). The space group Fd3m includes the spinel structure. 56 atoms, 32 oxygen anions distributed throughout a cubic closely packed structure, and 24 cations occupying 8 of the 64 tetrahedral sites (Asites) and 16 of the 32 octahedral sites (B sites) make up the cubic unit cell (Ellerby et al.).

Lead nanoferrites are successfully produced in this work using the hydrothermal and sol-gel methods. Various wet chemical processes, including as hydrothermal, sol-gel, micro-emulsion, chemical co-precipitation, etc., can be used to synthesise ferrites in nanocrystalline form. We employed the sol gel and hydrothermal routes, which offer significant benefits over the other approaches, out of all of them for the production of nanoferrite. Due to their ease of use, affordability, and the improved morphology of the synthesised Nanoferrites, these two approaches have gained prominence. These techniques can be used to create crystalline structures under high vapour pressure from aqueous liquids. Lead Nanoferrites with a variety of morphologies are produced using these techniques by controlling the development of particles and the recrystallisation of solution. Additionally, these techniques are employed to achieve enhanced characteristics, increased homogeneity, and a narrower particle dispersion, which affect the ferrites' structural, electrical, and magnetic properties (Ohshima et al.; Vollath).

The structural, optical, morphological, and magnetic characteristics of lead Nanoferrites that were produced by two different processes may be compared with this type of examination. Although some research has examined the characteristics of lead Nanoferrites produced through particular processes, there aren't many thorough comparisons between various production methods. The majority of research now in existence concentrate on a single synthesis technique without considering how several methods might affect the characteristics. There is a lack of research on the relationship between morphological properties (particle size, shape, and surface area) and optical characteristics (band gap, absorption) of lead Nanoferrites. Existing studies often treat these aspects in isolation, without sufficiently investigating how changes in morphology resulting from different synthesis methods might influence optical behavior. The main significance of this research is

Correlation of Properties: To Correlate the structural, optical, morphological, and magnetic properties to understand the impact of the synthesis method.

Effectiveness of Methods: To Conclude which method is more effective for producing lead nanoferrites with desired properties for specific applications.

This study could fill this gap by providing a side-by-side comparison, helping to determine the most effective synthesis method for specific applications. Filling this gap could enhance the understanding of how to control the optical properties of lead Nanoferrites through morphological tuning, which is critical for applications in optoelectronics and photonics.

Experimental Methods

Sol Gel Method

Using this technique, low-temperature synthesis of glass, ceramics, and other materials is prepared. In order to create a homogenous mixture, the precursors, [$Pb(NO_3)_2$] and [$Fe(NO_3)_3.9H_2O$], were first dissolved in 200ml of distilled water using a magnetic stirrer for three hours. 20ml of NaOH, which serves as a capping agent, is added after three hours of continuous stirring. Next, 20 ml of distilled water is added. The mixture is heated to 80^{o}C for two hours, and it is then dried for twenty-four hours at 80^{o}C. The product is gathered and reduced in size to a fine powder using a mortar. The last sintering was done for three hours at 600^{o}C, and then it was gradually cooled to room temperature at the same pace as healing. The pH level and reaction time for synthesizing lead Nanoferrites can vary depending on the specific synthesis method used, such as sol-gel and hydrothermal techniques. The pH has an impact on the stability and gel formation during sol-gel synthesis. To achieve uniform particle sizes and improved control over the final structure, the hydrolysis and condensation rates of the precursors are generally controlled at a pH between slightly acidic and neutral which is 7. There are several steps involved in the synthesis of sol-gel, such as gel formation, drying, and calcination. It took 24 hours to achieve the required final Nanoferrites qualities.

The following flow chart illustrates the preparation procedure.

Preparation Methods

PRECURSORS: Lead Nitrate [$Pb(NO_3)_2$],
Ferric Nitrate [$Fe(NO_3)_3.9H_2O$]

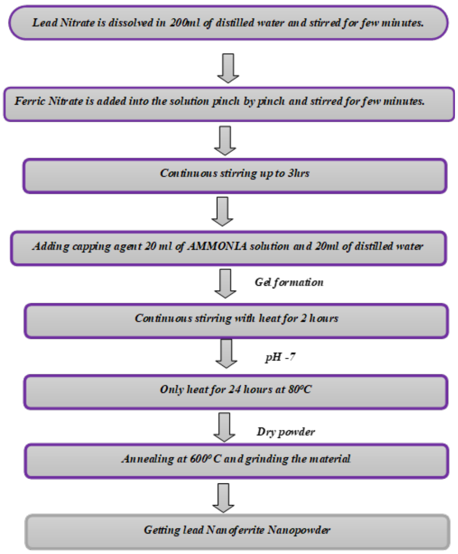


Figure 1 Flowchart for Lead Nanoferritenanomaterials by sol-gel method

Flow Chart For Hydrothermal Method

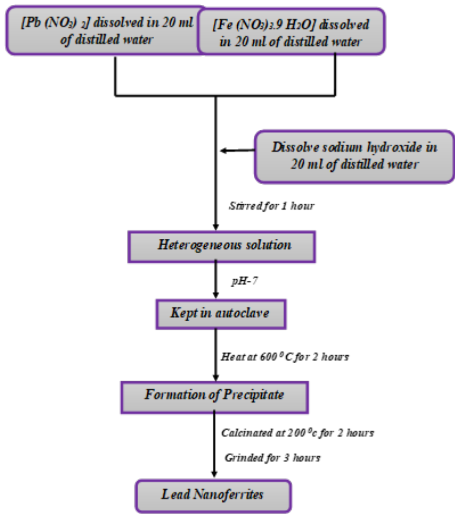


Figure 2 Flowchart for Lead Ferrite Nanomaterials by Hydrothermal Method

Hydrothermal Method

In order to create Lead Nanoferrites, stoichiometric molar quantities of ferric nitrate [$Fe(NO_3)_3.9H_2O$] and lead nitrate [$Pb(NO_3)_2$], were dissolved in 40ML of distilled water to create a transparent solution. The reactants were then combined using a magnetic stirrer until they were fully dissolved. NaOH was added to the mixture while stirring in order to function as a surfactant that coats the nanoparticles and keeps them from aggregating together. NaOH is dissolved in 20 millilitres of distilled water. A homogenous solution was achieved after two hours of constant stirring, and two thirds of the total volume was autoclaved. The pH in hydrothermal synthesis is adjusted depending on the desired phase and crystallinity. A neutral to basic pH is generally preferred to promote the growth of well-defined crystalline Nanoferrites. Here it is maintained between 7 to 8. Finally, the autoclave was allowed to cool to room temperature after being heated to 600°C for two hours. Hydrothermal synthesis generally requires longer reaction times which is 24 hours to ensure complete crystallization of the Nanoferrites. The extended time under high-temperature and high-pressure conditions allows for the growth of highly crystalline and uniform particles. To dry the obtained precipitate, it is annealed for two hours at 200°C. To create a fine powder, solid phase samples were taken and ground in a mortar. The powders that were obtained were utilised in the following measurements.

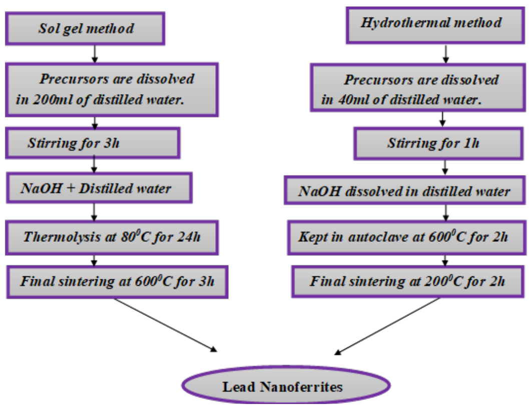


Figure 3 Flowchart for Comparison of Both Methods

Under equilibrium condition, chemical reaction can be expressed as follows:

$Pb(NO_3)_2 + 2Fe(NO_3)_3.9H_2O \rightarrow PbFe_2O_4 + 8NO_2 + 18H_2O + 2O_2$

Results and Discussion

Crystalline Structure

The lead XRD pattern Figure 4 shows nanoferrite that was created using two distinct methods. The broad diffraction peaks signify extremely small nanocrystalline size, whereas the strength of XRD peaks shows the crystalline nature. The sample made using the hydrothermal approach has the lowest lattice parameter, 9.43 A°, whereas the sample made using the sol gel method has the highest value, 9.805 A°. With α=β=γ=90°, only cubic structure was found for both approaches. The average crystallite size can be calculated using Debye Scherrer's formula by determining the full width at half maximum (FWHM) of the matching maximum intensity peak.

$$D = K\lambda / (\beta\cos\theta) \qquad (1)$$

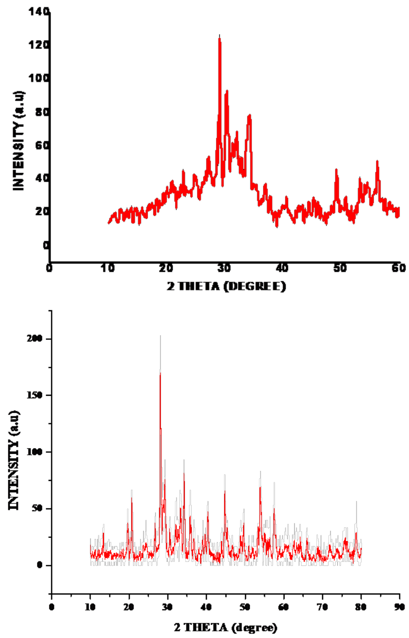
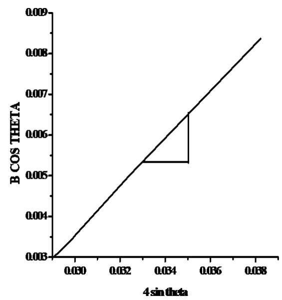


Figure 4 Lead Ferrite XRD Patterns Produced using the Sol Gel and Hydrothermal Methods

In this equation, K is typically assumed to be 0.89 for spherical nanoparticles, D is the crystallite size, is the X-ray radiation wavelength, and is the line width at half maximum height. For lead ferrite synthesised using sol gel and hydrothermal methods, the crystalline sizes are found to be 33 nm and 20 nm, respectively.



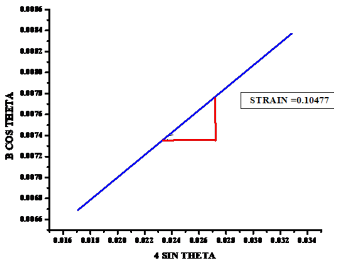


Figure 5 Williamson's Hall Plot for Lead Ferrite by Sol Gel and Hydrothermal Method

The length of a dislocation line per unit volume of synthesised nanomaterials is known as the dislocation density. The dislocation densities (δ) of lead ferrite synthesised via sol gel and hydrothermal technique are $2.4554x10^{15}$ m^{-2} and $2.3302x10^{15}m^{-2}$, respectively, as determined by the following relationship.

$\delta = n/D^2$

where, D is a crystallite size and n=1. The strain (ε) value is calculated as 0.24409 and 0.148119 using Williamson- Hall plot for the synthesized nanomaterials by sol gel and hydrothermal method respectively as shown in figure 5. The different physical parameters obtained from XRD are tabulated as shown.

Table 1 Physical Parameters from XRD Analysis

Method	Crystallite size	Strain	Dislocation density
Sol Gel method	33nm	0.24409	$2.4554x10^{15}$ m^{-2}
Hydrothermal method	20nm	0.14811	$2.3302x10^{15}$ m^{-2}

Fourier Transform Infrared Spectroscopy

A good way for examining the vibrational characteristics of the molecules found in the synthesised materials is FTIR Spectroscopy. Between 400 and 4000 cm^{-1} is the wave number range in which the vibrational spectra is captured. FTIR spectra are employed for identifying various functional groups and chemicals. As seen in figure (6) below, the peaks are visible in the vibrational spectra, which display the various functional groups present in$PbFe_2O_4$.

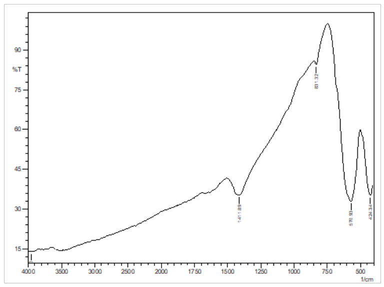


Figure 6 FTIR Spectrum of Lead Ferrite Synthesized by Sol Gel Method

From the FTIR spectrum, at 1411 cm^{-1}, C-C stretch occurs corresponding to aromatics. The peak at $831.32cm^{-1}$, represents the C-Cl stretch with alkyl halides. C-Br stretch is present in $570.93cm^{-1}$, represents the alkyl halides. Also, the presence of peak at the wave number $424.34cm^{-1}$ confirms the existence of alkyl halides with C-I stretch.

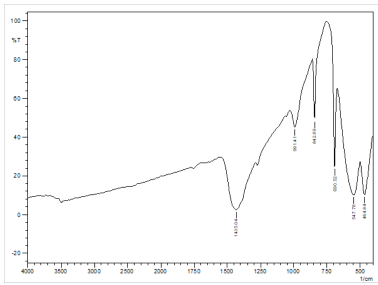


Figure 7 FTIR spectrum of lead ferrite synthesized by hydrothermal method

The different functional groups are identified from the FTIR spectrum. They reveal that the transmittance peaks at $1435.05cm^{-1}$ represents the C-C stretch with aromatics. At $991.41cm^{-1}$ occurs C-H bending. Also, C-Cl stretch occurs at $842.89cm^{-1}$ which resembles alkyl halides. The peak at $547.78cm^{-1}$ confirms the C-Br stretch in alkyl halides group. C-I stretch is observed at $464.81cm^{-1}$.

UV-Visible Spectral Study

To determine the optical absorption of the sample, the UV-visible absorption spectra is examined at room temperature in the 200-1200 nm range.

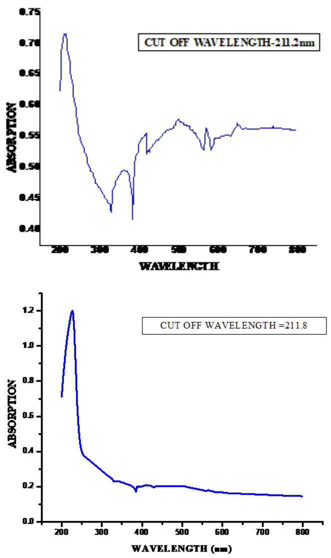


Figure 8 Cut-off Wavelengths of Lead Ferrite by Sol Gel and Hydrothermal Method

The figure 8 shows the cut-off wavelength of lead ferrite nanomaterials synthesized by sol gel and hydrothermal methods respectively. From the graph, the cut-off wavelength starts from 211 nm and it is found to be in the visible region. The optical transmittance and band gap energies is,

$(\alpha h\upsilon)^n = A\ (hv\text{-}Eg)$

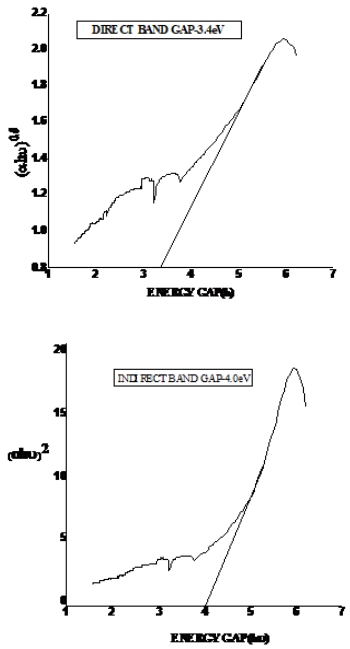


Figure 9 Direct and Indirect Band Gap for Lead Ferrite Synthesized by Sol Gel Method

The material's optical band is represented by Eg, an energy-independent constant, and the type of transition determines the exponent n. For an indirect transition, the value of n is an integer; for a direct transition, it is a half-integer. A plot of $(\alpha h\upsilon)^n$ Vs $h\upsilon$ is plotted to determine this. Figure 9 displays the Tauc plots for the direct and indirect band gap transitions for lead ferrite that was synthesised using the sol gel technique. The graphs show that the energy gaps for the direct and indirect transitions are, respectively, 3.4eV and 4 eV.

The Tauc plot is plotted for both direct and indirect band gap transition for lead ferrite synthesized by hydrothermal method is shown in figure 10 respectively. From the graphs, it is observed that energy gap for direct transition is 4.6eV and for the indirect transition, it is 5eV respectively.

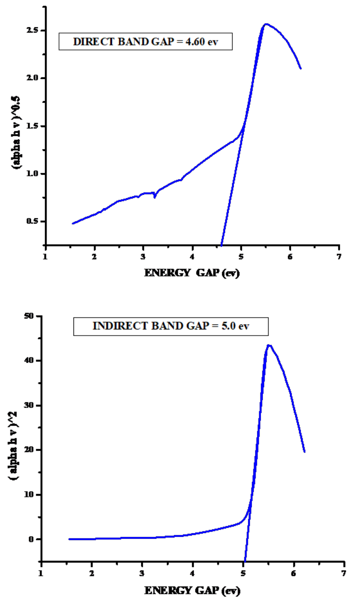


Figure 10 Direct and Indirect Band Gap for Lead Ferrite by Hydrothermal Method

Scanning Electron Microscopy(SEM)

Microstructural and morphological investigations were conducted by the use of a scanning electron microscope.

Figure 11 and Figure 12 shows the SEM images of lead ferrite nanomaterials prepared by Sol gel method shows nanoflakes in the magnification range of 1µm, 5µm and 500nm and hydrothermal method shows nanoparticles in the magnification range of

5 µm, 10 µm and 20 µm respectively. The Lead Nanoferrites are distributed regularly on the whole surface and it also reveals that the sample contains smaller crystallite size which is less than 100nm.

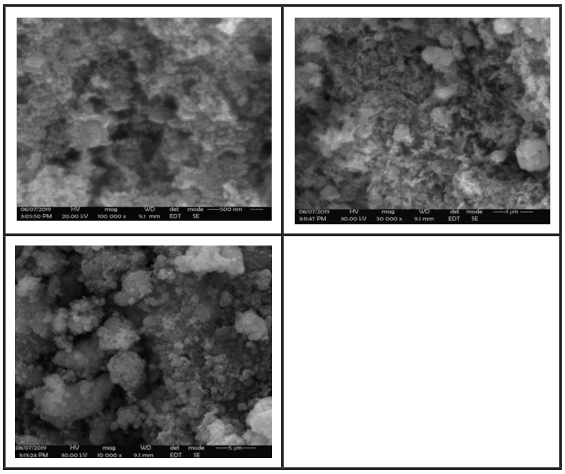


Figure 11 SEM Images of Lead Ferrite Prepared by Sol Gel Method

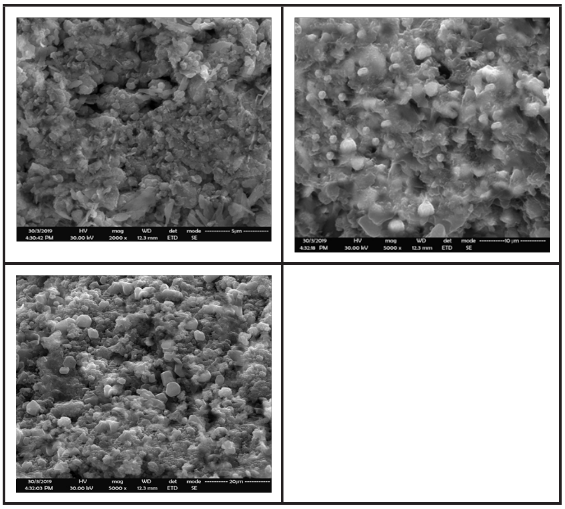


Figure 12 SEM Images of Lead Ferrite made using a Hydrothermal Process

Vibrating Sample Magnetometer (VSM)

Vibrational Sample Magnetometer (VSM) was used to record the hysteresis loop of the sample produced by the sol gel and hydrothermal process in order to understand the magnetic properties of lead ferrite. The loop has been used to determine the magnetic characteristics, including saturation magnetisation, coercivity, and sensitivity.

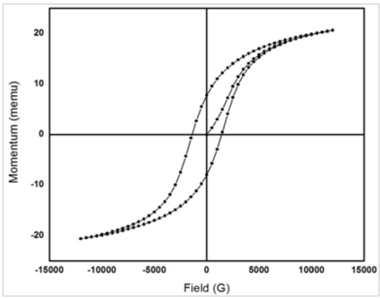


Figure 13 Hysteresis Loop of Lead Ferrite Synthesized by Sol Gel Method

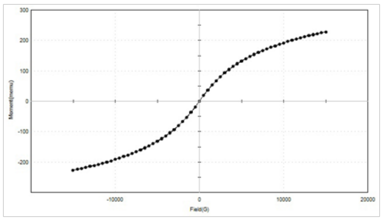


Figure 14 Hysteresis Loop of lead ferrite synthesized by hydrothermal method

The magnetic parameter such as magnetization is 0.2064emu and 0.3359emu, the retentivity is 7.8964emu and 13.5045emu and the Coercivity is 1413G and 0.4428G respectively. On comparing the hysteresis loop of the synthesized Nanoferrites, the lead ferrite synthesized by hydrothermal method finds its application as there is no loses during magnetization and demagnetization of the material, whereas the same sample synthesized by sol gel method undergoes some loses during magnetization and demagnetization of the material. The lead nanoferrites synthesized by hydrothermal method have zero Coercivity hence they are said posses super paramagnetic property.

Conclusion

Lead ferrite nanomaterials have been successfully synthesized by sol-gel and hydrothermal method

- According to XRD data, Lead Nanoferrite produced by Hydrothermal synthesis has smaller crystallite size, strain, and dislocation density than that of the Sol gel approach.
- Using FTIR spectrograph, the different functional groups and molecular vibrations are conformed which shows the presence of the substituted metal ions.

- From UV-VIS spectroscopic studies, the cut-off wavelength is found to be 211.2nm and 211.8nm respectively.
- The optical band gaps are calculated using Tauc plot and the direct band gap is found to be 3.4eV and 4.6eV and the indirect band gap is found to be 4eV and 5eV.
- The morphological survey by SEM analysis shows nanoflakes for sol gel sample at magnification range of 1µm, 5µm and 500nm and shows nanoparticles in the magnification range of 5 µm, 10 µm and 20 µm respectively.
- From VSM results, magnetic properties of synthesized nanomaterials are analyzed. It is clear that the Nanoferrites synthesized by hydrothermal route have zero Coercivity and finds applications in transformer and motor cores to minimize the energy dissipation and also in some electronic devices.

From this work we found that the results obtained from all the characterization techniques, the Lead Nanoferrite particles synthesized through hydrothermal have very good results leading to highly challenging nanomaterials of very low dimension. To mention the particle size, strain and dislocation densities are decreased in hydrothermal method. Also, from the SEM analysis it has observed that uniform formation and distribution of nanoparticles rather than sol-gel method. Furthermore, from the VSM analysis the same material synthesized through hydrothermal method achieves super paramagnetic behavior which is less pronounced in the material synthesized by sol-gel method.

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