

Investigation of Structural, Morphological and Optical Characteristic of Co-precipitated ZnFe_2O_4 Nanoparticle

Zaki Nawaz*

Department of Physics Faculty of Sciences The Superior University Lahore Pakistan
Email: chzaki282@gmail.com

Muhammad Rizwan Aslam

Department of Physics Faculty of Sciences The Superior University Lahore Pakistan

Muhammad Arslan Naseer

Department of Physics Faculty of Sciences The Superior University Lahore Pakistan

Thiraj Ahmed

Department of Physics Faculty of Sciences The Superior University Lahore Pakistan

Ayesha Ashraf

Department of Physics Faculty of Sciences The Superior University Lahore Pakistan

Sabir Abbas

Department of Physics University of Sargodha, Punjab, Pakistan

ABSTRACT

Zinc Ferrite (ZnFe_2O_4) nanoparticles have gained high interest due to their unique structural, morphological, vibrational behavior and optical response which make them appropriate materials for range of advanced technological applications. In this study ZnFe_2O_4 nanoparticles were prepared through co-precipitation method. Zinc chloride (ZnCl_2) and ferric chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) used as the precursor salts and precipitating agent sodium hydroxide (NaOH) respectively. The synthesized material was systematically characterized to study the structural, morphological, chemical bonding and optical behavior. The (XRD) pattern indicate the formation of cubic spinel phase of ZnFe_2O_4 . The characteristic diffraction peaks that occurred at (220), (311), (511) and (440) crystallographic planes. The crystallite size of the synthesized material is calculated from the strongest diffraction peak by means of the Debye–Scherrer formula is around 13.24 nm that supports the nanocrystalline character of the material. The formation of the ferrite lattice was evidenced by the presence of characteristic vibrational bands at ~ 482 and 772 cm^{-1} as confirmed by (FTIR). The morphology and surface features of the created nanoparticles were examined by (SEM) technique. The optical properties of sample was investigated by the UV–Visible spectroscopy and its optical band gap was calculated the Tauc method. The band-gap energy obtained is about 2.2 e v which can be correlated with semiconducting property of ZnFe_2O_4 and its capacity to absorb the visible light

Keyword: (ZnFe₂O₄), Zinc Ferrite, Zinc Chloride, Ferric Chloride, Co-Precipitation, XRD, FTIR, UV, SEM Band-Gap Energy.

Introduction

The field of nanotechnology has been an interesting branch of materials science because of the extraordinary contrast in the physicochemical properties of materials produced at the nano-scale vs their bulk state. The modification of the size-dependent electronic effects, the high surface to volume ratio and altered structural properties of nanomaterials are the most important changes associated with them. Hence, metal oxide nanoparticles have received considerable attention in various advanced applications such as photocatalysis, sensing, energy conversion and storage, environmental remediation and so on[1]. Spinel ferrites constitute an important class metal oxide material because of their structural, magnetic, electrical, optical, chemical and thermal stability, have been of particular interest among the metal oxide nanomaterials. Spinel ferrites commonly represented by formula (MFe₂O₄) in which M denotes a divalent metal ion such as Zn²⁺, Ni²⁺, Co²⁺, Mn²⁺, Mg²⁺ or Cu²⁺[2]. They are highly controlled by the distribution of the cations between tetrahedral (A) and octahedral (B) sites within the oxygen based crystal lattice. The distribution of cations, crystallite size, crystallite shape and defects in the crystal structure could, therefore, lead to a significant change in the functional properties of ferrite nanoparticles[3].

Zinc ferrite (ZnFe₂O₄) is widely studied members of the spinel ferrite family because of its optical tunability, semiconducting properties, low toxicity and chemical stability. Normally, the ZnFe₂O₄ in bulk form carry typical spinel arrangement with Zn²⁺ cations occupying tetrahedral sites and Fe³⁺ cations occupying octahedral sites[4]. At the nanoscale, however, partial cation inversion does occur which results in redistribution of Zn²⁺ and Fe³⁺ cation among available lattice sites. The changes in structure can significantly affect the crystallinity, particle morphology, electronic structure, magnetic properties and optical properties of ZnFe₂O₄. Therefore, it is very important to control the synthesis conditions to meet the requirement of desirable structural and functional properties of nanoparticles[5].

Synthesis method and processing conditions have a strong impact on the properties of the nanoparticles made of ZnFe₂O₄. They have been prepared using several methods such as sol-gel, hydrothermal, combustion, solvothermal, thermal decomposition and co-precipitation. Of those methods, the co-precipitation method is especially interesting due to its simplicity, low cost, relatively low processing temperature and uniformity of composition and its applicability to the fabrication of nanocrystalline ferrites[6].

Although zinc ferrite nanoparticles have been widely investigated, the structural and optical response are still very sensitive to the synthesis method and the resulting microstructure of the nanoparticles investigated. Therefore, it was crucial to systematically correlate the structure, morphology, vibration and optical properties of sample prepared with a particular synthesis method to understand its behavior[7].

The purpose of present work is to prepare ZnFe₂O₄ nanoparticles by using zinc chloride and iron chloride as precursor salts and precipitating with sodium hydroxide and heat treatment. (XRD), (SEM), (FTIR) spectroscopy and UV-Visible

spectroscopy were used to systematically describe synthetic material[8]. Structural phase, crystallite size, surface morphology, metal–oxygen bonding and optical captivation behavior and optical band gap of synthesized nanoparticles of ZnFe_2O_4 was investigated to establish the relationship between the structural and optical properties of the prepared nanoparticles of ZnFe_2O_4 . The study provides a detailed evaluation of the ZnFe_2O_4 prepared by simple co-precipitation technique, and a relationship between the structure and properties[9].

Materials and methods

Materials

Zinc ferrite nanoparticles were synthesized using commercially sourced chemical reagents. Zinc chloride, ferric chloride hexahydrate and sodium hydroxide had purity of 99.9%. Those were used as obtained and of analytical grade.[10]

Synthesis of ZnFe_2O_4 Nanoparticles

The co-precipitation process used to prepare the nanoparticles of (ZnFe_2O_4). The clear precursor solutions prepared by dissolving of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (4.0 g) and ZnCl_2 (2.4 g) in 30mL of distilled water with continuous magnetic stirring. Iron precursor added slowly drop by drop to the zinc precursor and stirring constantly to get homogeneous mixing. Dark precipitate was formed by adding an aqueous NaOH solution dropwise with stirring to the solution until the pH reached 9-10. This suspension was continuously stirred at 90°C for 2hours[11]. The precipitate was collected by filtration and repeatedly washed with distilled water until the filtrate reached at neutral pH (pH 7). A precipitate was formed in the wash and dried at 80 °C – when dry it was ground in a mortar and pestle to obtain a fine powder. The powder was finally annealed for 2 h at 700 °C in a muffle furnace to induce crystallization and formation of the ZnFe_2O_4 spinel structure. The resulting ZnFe_2O_4 powder was allowed to cool down to room temperature and the structure, morphology, vibration and optical properties of powder studied[12].

Characterization

To explore the structural, morphological, vibrational, optical properties of prepared ZnFe_2O_4 nanoparticles, characterization of nanoparticles done using (XRD), (SEM), (FTIR) spectroscopy, and UV [13]. Composition of crystalline phase and the degree of crystallinity of the material were also determined by X-ray diffraction and the average crystallite size derived from the primary diffraction peak via the Debye–Scherrer formula. The characterization of synthesized nanoparticles was done by SEM to understand the surface morphology, surface distribution and aggregation of nanoparticles. FTIR spectra were recorded 400 to 4000 cm^{-1} range because the characteristic metal–oxygen vibrations are expected in that spectral range and to explore the chemistry of the ferrite's bonding structure[14]. UV–Visible spectrophotometry used to assess the light absorption property by scanning the wavelengths around 200–800 nm. Optical band gap value determined by applying Tauc model for directly allowed transition: $(\alpha h\nu)^2 = A(h\nu - E_g)$, where α shows absorption coefficient, $h\nu$ represents photon energy, A is a proportional constant and E_g indicate optical band gap energy[15].

Results and Discussions

XRD Analysis

For the confirmation of the formation and the crystalline nature of synthesized ZnFe_2O_4 nanoparticles, the X-ray diffraction (XRD) technique was used in the current research. The characteristic planes of a cubic spinel structure were determined by analysis of diffraction peaks and can be computed numerically based on a peak's FWHM using the Scherrer equation. Only when D is the crystallite size, K is the crystallite shape factor (≈ 0.9), λ is the X-ray wavelength, β is the FWHM in radians, and θ is the Bragg angle can these equations be used[16].

The XRD pattern of the synthesized nano ZnFe_2O_4 is presented in figure 3.1. The diffraction peaks observed at approximately 30° , 35.3° , 57° , and $62\text{--}63^\circ$ can be indexed to the crystallographic planes (220), (311), (511), and (440), in that order. The reflections are consistent with the reported data in the literature and are typical of the cubic spinel structure of ZnFe_2O_4 [17]. The high intensity of the (311) reflection is a special feature of the spinel phase ZnFe_2O_4 . Clear and relatively sharp diffraction peaks suggest that the synthesized material is quite crystalline. Moreover, there were no additional diffraction peak observed in the diffraction pattern which can be assigned to other phases, indicating that material is mainly single crystalline ZnFe_2O_4 [14].

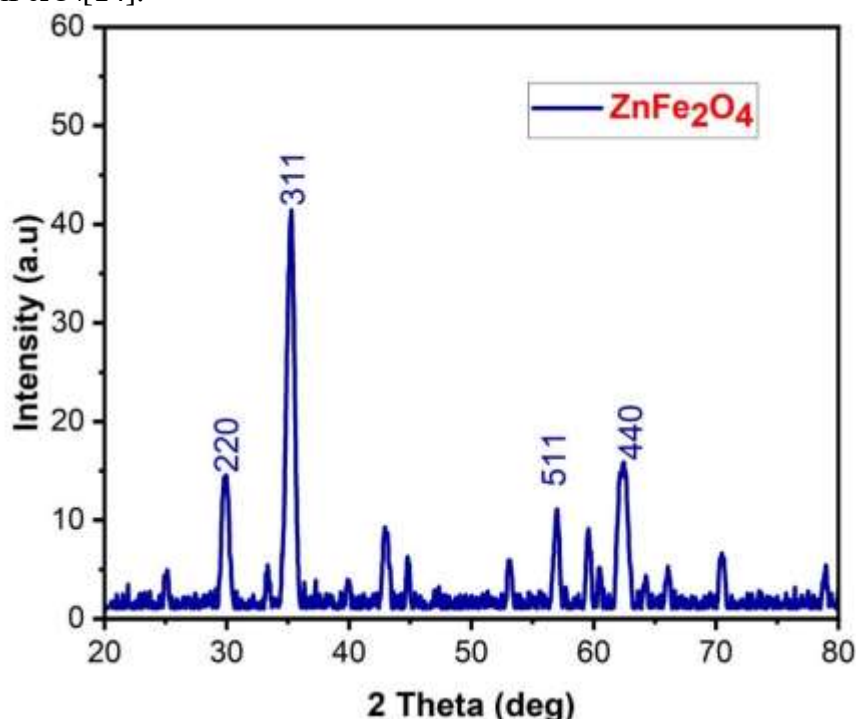


Figure 1: XRD spectrum of zinc ferrite (ZnFe_2O_4).

FTIR Analysis

The chemical binding of the formed ZnFe_2O_4 nanoparticles and the vibration properties were studied with Fourier Transform Infrared (FTIR) spectrometer. The FTIR spectrum of the synthesized ZnFe_2O_4 was obtained in the range of $4000\text{--}400\text{ cm}^{-1}$ and the observed peaks were analyzed to confirm the spinel ferrite structure[18].

The most important FTIR data for ZnFe_2O_4 is found in the lower wave number region which corresponds to the vibration of metal–oxygen (M–O) bonds. This band of 482 cm^{-1} is of special interest because it is in the characteristic region of vibration of metal–oxygen bonds in spinel ferrites. Literature data indicate that, for the case of ZnFe_2O_4 , the low frequencies associated with the M–O vibrations are associated with the octahedral sites[19].

Figure 3.2 shows the synthesized nanoparticles of ZnFe_2O_4 with typical characteristics of the lattice vibration of the ferrite lattice. The absorption band at 482 cm^{-1} is probably caused by the metal–oxygen stretching vibrations that take place in the octahedral positions of the spinel structure. The band at 772 cm^{-1} might be related to lattice/microstructural vibrations of ZnFe_2O_4 . The weak/broad in the high wave number region may be associated to be connected to the hydroxyl groups on the surface or to water molecules that have been adsorbed there. Metal-oxygen vibrations are present in the low wave number region is encouraging for the presence of the spinel ferrite structure of the ZnFe_2O_4 [20].

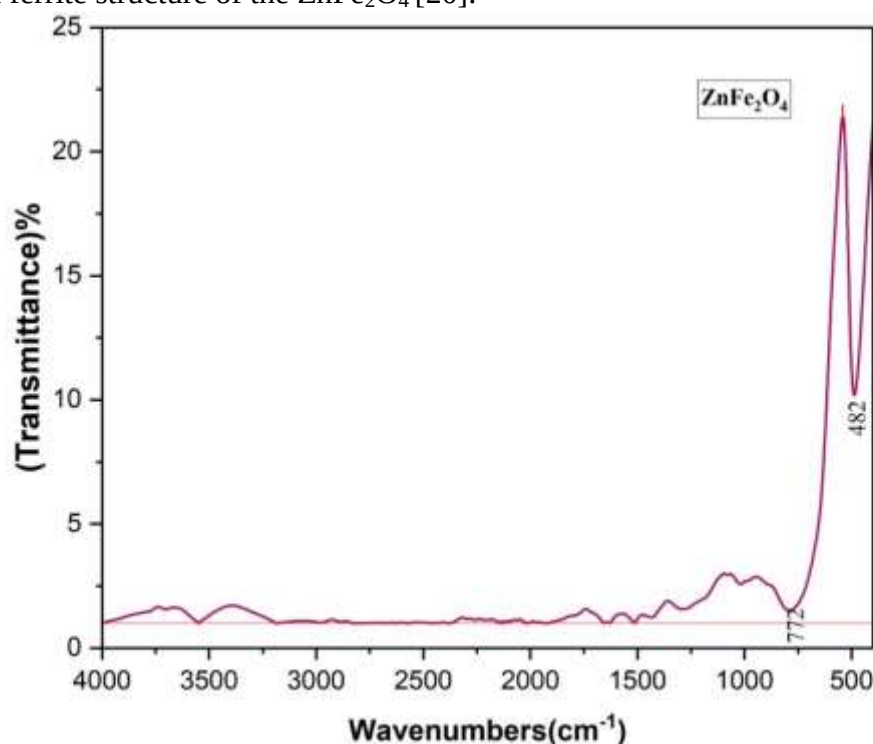


Figure 2: FTIR analysis of zinc ferrite (ZnFe_2O_4).

UV–Visible Analysis

UV–Visible spectroscopy has been used to learn synthesized ZnFe_2O_4 nanoparticles for optical properties. The Absorption behaviour was studied with respect to the photon energy ($h\nu$) whose value is calculated from the measured wavelength by equation: $h\nu = E = \lambda / hc$, where, h is Planck's constant, c is the speed of light and λ is the wavelength of the absorbed radiation. The optical band gap of the optical was estimated by using the Tauc relation which assumes a direct allowed gap, $(\alpha h\nu)^2 = A(h\nu - E_g)$. In this equation, α is the absorption coefficient, (A) is a proportionality

constant and $(h\nu)$ is the energy of the photon and (E_g) is the optical band gap[21]. Thus, graph of $(\alpha h\nu)^2$ against $(h\nu)$ drawn. The straight line part of absorption edge was extrapolated on the photon energy axis and the point of extrapolation was considered as the optical energy gap. Optical band gap of ZnFe_2O_4 nanoparticles synthesized found to be ~ 2.2 eV, as obtained from the Tauc plot indicating that the synthesized nanoparticles possess semiconducting properties with a greatly increased absorption of light in the visible spectrum, which is the area of interest for practical applications. The found direct gap of the ZnFe_2O_4 nanoparticles is ~ 2.2 eV. The obtained band gap values are in line with other reports in the literature that state that pure ferrites have a band gap of 1.9–2.5 eV. The electron confinement is responsible for the observed band gap expansion of ZnFe_2O_4 nanoparticles. The electron excitation from the O 2p level into the Fe 3d level in normal spinel, like Zn^{2+} and Fe^{3+} cations are located in tetrahedral and octahedral sites, respectively. The valence band and the conduction band of ZnFe_2O_4 are usually considered as the O-2p orbital and the Fe-3d orbital, respectively. The electron excitation from the O 2p level into the Fe 3d level may be the cause of ZnFe_2O_4 absorption in the visible light region[22].

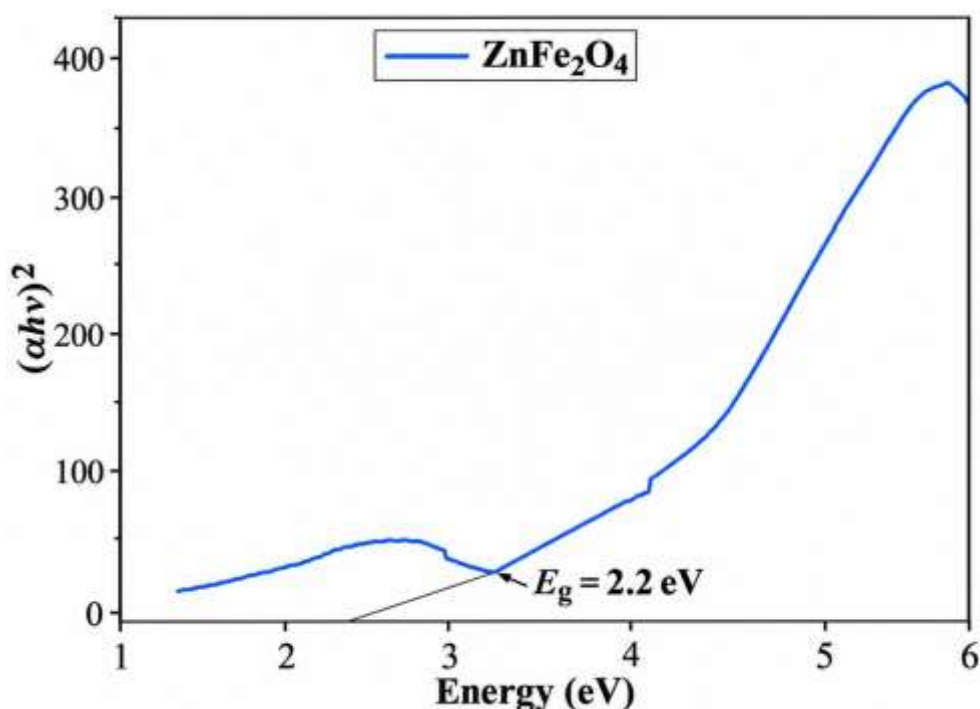


Figure 3: Tauc plot of the synthesized zinc ferrite (ZnFe_2O_4).

SEM Analysis

Scanning electron microscopy (SEM) was used to examine the surface morphology of the produced ZnFe_2O_4 nanoparticles at the magnification of $\times 2500$ and $\times 10000$. Morphology is heterogeneous as seen in micrographs, and is highly agglomerated with irregular granular, angular and quasi-spherical particles in some places. With higher magnification, it can be seen that there are finer particles in these aggregates, and relatively large aggregates can be seen at lower magnification[23]. The high

surface energy of the nanoparticles, van der Waals and magnetic interactions between particles, and the impact of the drying and annealing processes during the synthesis are the primary causes of the observed agglomeration. Based on SEM results, the nanoparticles are not completely isolated, but are in the form of close aggregates. It would be noted that the size measured by SEM could be that of an agglomerated particle[9].

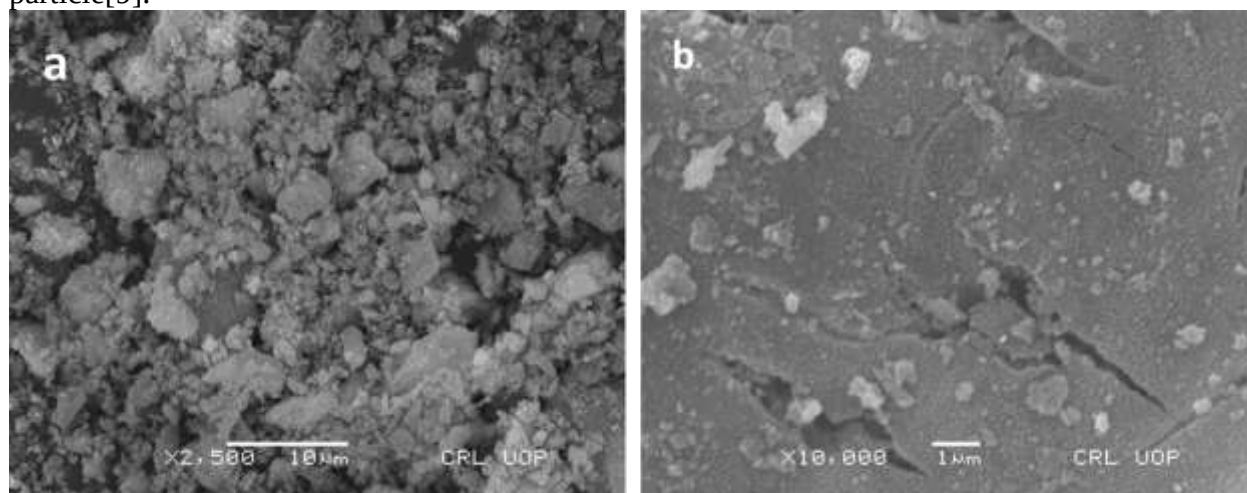


Figure 4(a,b):Shows the morphology and agglomeration of zinc ferrite (ZnFe_2O_4).

Conclusion

ZnFe_2O_4 nanoparticles had been synthesized by using the co-precipitation process with zinc chloride and iron chloride as the precursor salts followed by precipitating with NaOH. The XRD analysis confirmed the spinel phase in the form of cubic ZnFe_2O_4 with (220) (311) (511) and (440) planes. The average crystallite size was calculated to be 13.24 nm by Debye–Scherrer equation. The characteristic metal-oxygen vibrations in the FTIR spectra at around 482 and 772 cm^{-1} confirmed the formation of spinel ferrite lattice. SEM analysis showed a very agglomerated and heterogeneous morphology. The synthesized material was found to be semiconducting with an optical band gap of ~ 2.2 eV from the UV–Visible analysis. Overall, the results showed the successful formation of nanocrystalline ZnFe_2O_4 having different structural, morphological, vibrational, and optical properties, which would be useful for its use as a functional nanomaterial.

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Online ISSN

3007-3197

Print ISSN

3007-3189

<http://amresearchreview.com/index.php/Journal/about>

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