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Structural, Optical and Photocatalytic Study of Cobalt doped Zinc Ferrite

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Abstract

The polycrystalline powder of $Zn_{1-x}Co_xFe_2O_4$ with a variable Co concentration ranging from 0.0 to 0.3 was created using the Sol Gel assisted auto combustion process. The powder X-ray diffraction technique was used to investigate the sample's structural phase identification. The synthesized ferrite material cubic structure with Fd3m space group was shown by X-Ray Diffraction data. The unit cell volume and lattice parameter values have changed slightly as a result of the doping of Co ions, but the crystal structure remains unchanged. In the X-Ray Diffraction data, no additional peaks are observed. In Diffuse reflectance spectroscopy (DRS) shows The band gap of $Zn_{1-x}Co_xFe_2O_4$ ($x = 0.0 - x = 0.3$) are found to be 3.10eV, 3.07eV, 2.98eV, and 2.92eV Respectively. As Co concentration increases band gap goes on decreasing. Band gap decreases ($x=0.0$ to $x=0.3$) that leads to increasing the photocatalytic performance. In photocatalytic degradation as Co concentration increases percentage of degradation increases. The % of Degradation ~ 45% at $x=0.0$ as Co concentration increases % of degradation also increases up to the ~73% at $x = 0.3$ Respectively. The work demonstrates the potential of co-doped zinc ferrite as a powerful photocatalyst driven by visible light.

Keywords: Zinc ferrite; Spinel Ferrite Co-doping, XRD, UV-DRS, Band Gap, Photocatalysis, Methylene Blue, Nanomaterial

Introduction

Ferrites are a kind of ferromagnetic materials. [1] ferrites are gaining a lot of attention in scientific research because of their distinct electrical and magnetic properties. There are various application of ferrites such as high-frequency electronics, biomedical fields, wastewater treatment, magnetic storage devices, sensors, and photocatalysis. Researchers are becoming more interested in magnetic nanoparticles because of their unique magnetic and electrical properties. [2-3] The spinel ferrites group of ferrites are grown in various fields. The typical formula for spinel is MFe_2O_4 , where M is an ion of a divalent metal. The properties of spinel ferrite changed by doping and the distribution of cations inside the molecule. The cubic crystal structure of spinel ferrites is closed-packed arrangement of metal cations and oxygen ions scattered at tetrahedral (A) and octahedral [B] sites. [4-5]. The chemical mixture of cation distribution at the (A) and [B] sites are the primary determinants of spinel ferrites [6]. Zinc ferrite have various characteristics, such as surface area, surface shape, crystallite size, defect level, optical properties, saturation magnetization, etc., and these characteristics can be tailored further improve structural, magnetic, and catalytic performance by dopants such as cobalt (Co) [7-8]. In Catalysis and wastewater treatment, spinel ferrites are frequently used as magnetic adsorbents and their primary benefit is the ease with which they can be separated from aqueous solutions by applying an external magnetic field [9]. The synthesis method shows a crucial role in investigating the physicochemical properties of $ZnFe_2O_4$ nanoparticles. The desired particle characteristics have been achieved using a variety of techniques, including co-precipitation, hydrothermal synthesis, ball milling, sol-gel, and solid-state methods. Sol-gel auto combustion produces the most uniform and pure nanoparticles at the lowest temperature, since it produces nanoparticles of uniform size with regular microstructures. [10-16]. A sol-gel process creates a network by colloiddally suspending solid particles in a liquid to create a sol, which is then gelled into a three-dimensional structure. A rapid exothermic reaction occurs between precursor solution of metal salts and chelating agent (suitable fuels) in sol-gel assisted auto combustion, leading to the formation of the desired material. The gel is formed after the solvent evaporates and remaining particles begin to form interconnected networks after the solvent evaporates and Converting monomers into colloidal solution is called gel formation, that is, it is the formation of colloidal solutions. The end result of the process is colloidal powder films this process guarantees that the final product has a controlled microstructure and a consistent nanoscale dimension.

Furthermore, the low temperature employed makes the approach effective for synthesizing nanoparticles [17-18]. as used the sol-gel auto combustion approach to synthesize cobalt-doped zinc ferrite nanoparticles with $C_6H_8O_7 \cdot 2H_2O$ as a chelating agent. The synthesis method has several benefits, such as simplicity, scalability, and the ability to create nanomaterials with specific properties. Because of their various properties, the materials that are produced has variety of application such as energy storage, sensors, biomedical devices, and catalysis [21]. in the present study, Sol-Gel auto combustion process was used to prepare $Zn_{1-x}Co_xFe_2O_4$ ($x = 0.0, 0.1, 0.2$, and 0.3). This approach was chosen due to its low cost, low temperature and easy to handle and straightforward procedure.

Material and Methodology

1. Preparation of $Co_{0.3}Zn_{0.7}Fe_2O_4$

The starting materials were Zinc Nitrate $Zn(NO_3)_2 \cdot 6H_2O$, Cobalt Nitrate $Co(NO_3)_2 \cdot 6H_2O$, Ferric Nitrate $Fe(NO_3)_3 \cdot 9H_2O$ and citric acid $(C_6H_8O_7) \cdot 2H_2O$. The stoichiometric ratio of metal nitrates to citric acid is 1:1 maintained. The precursors were added in 50 ml distilled water and the solution were stirred in order to attain the homogeneous solution. Then the ammonia was mixed slowly maintaining the pH value equal to 7-8. After that the mixture was continuously stirred at $80^\circ C - 90^\circ C$ for 3 hours. The solution turned into gel when it was heated further. The resultant gel laterally expands into a fluffy mass resembling foam and then undergoes self-propagating combustion and turned to fine powder. The obtained powder was ground for 60 minutes and then it was annealed in a muffle furnace for four hours at $800^\circ C$.

Result and Discussion

2. X-Ray Diffraction

According to the Normalised XRD Pattern, the lattice planes (220), (311), (222), (400), (422), (511) and (440) were present at different 2θ values. The XRD pattern was used to determine the synthesized ferrite nanoparticle's single-phase cubic spinel structure. Strong reflection observed in (311) plane it indicate the spinel phase which belongs to the $Fd3m$ space group [24]. Particles with a broad XRD line indicate that they are nano sized. using Scherrer's equation, the average crystallite size of the samples was calculated. the average crystallite sizes varied between 35 and 45 nm. There was a match between the observed diffraction peaks and JCPDS card number #2210-12 based on their intensity peaks and their d values. Fig. 1 shows the XRD pattern for Co-Zn ferrite. The intense peak (311) is used to calculate the lattice parameter of the spinel ferrite. Zinc ferrite is enriched with Co, the lattice parameter of

$Zn_{1-x}Co_xFe_2O_3$ is reduced to 0.8418 nm, 0.8387 nm, 0.8382 nm, and 0.8304 nm, which is due to the smaller ionic radius of Co^{2+} (0.072 nm) than Zn^{2+} (0.074 nm). Table.1 shows the parameters calculated for the synthesized ferrite using the following equations.

Scherrer's equation accounts for peak broadening due to crystallite size (D). However, peak broadening also results from microstrain (ϵ) in the crystal lattice. The modified Scherrer equation, which includes both contributions, is given by [26],

$$\ln(\beta) = \ln\left(\frac{k\lambda}{D}\right) + \ln(1/\cos\theta)$$

Crystallite size is calculated by taking into account lattice strain contribution to peak broadening. Since the peak broadening is caused by micro-strain and crystal size, β_{hkl} expressed as follows:

$$\beta_{hkl} = \beta_{size} + \beta_{strain} \quad \text{--- (2)}$$

$$\text{But strain is } 4 \cdot \epsilon \cdot \tan\theta \text{ and size is } \frac{k\lambda}{D\cos\theta}$$

$$\therefore \beta_{hkl} = \frac{k \cdot \lambda}{D \cdot \cos\theta} + 4 \cdot \epsilon \cdot \tan\theta \quad \text{--- (3)}$$

$$\therefore \beta_{hkl} \cdot \cos\theta = \frac{k \cdot \lambda}{D} + 4 \cdot \epsilon \cdot \sin\theta \quad \text{--- (4)}$$

The micro strain in the sample is calculated using the formula $\epsilon = \Delta d / d$ [25-26].

X-ray density of material is given by the following formula,

$$\rho_x = \frac{8M}{Na^3} \quad \text{--- (5)}$$

where, M is molecular mass of the sample, N is Avogadro number and 'a' is the lattice parameter. By using sauter's formula to calculate the specific surface area of samples and this specific surface area is important for the heterogeneous catalysis, adsorption and surface reactions.

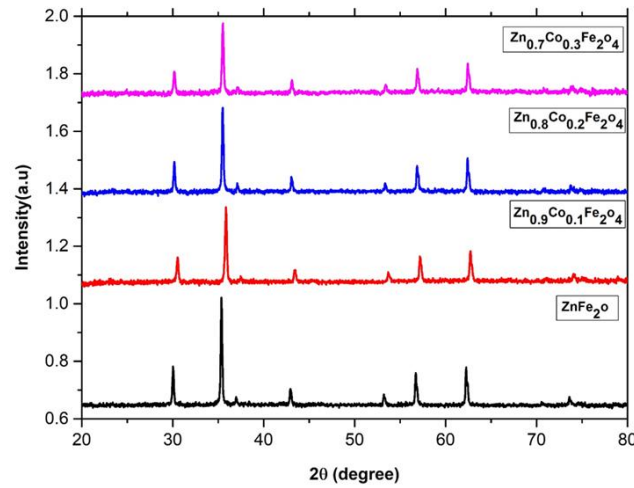


Fig. 1 XRD Plot $Zn_{1-x}Co_xFe_2O_4$ ($x = 0.0, 0.1, 0.2, 0.3$)

Table 1: Structural parameters of $Zn_{1-x}Co_xFe_2O_4$ ($x = 0.0, 0.1, 0.2, 0.3$) as obtained from X-Ray diffraction Analysis.

| Parameters | Co Concentration | | | |
|---|------------------|--------|--------|--------|
| | 0.0 | 0.1 | 0.2 | 0.3 |
| $V_{cell} (Å^3)$ | 594.31 | 570.85 | 587.86 | 586.96 |
| Crystalline Size(D) (nm) Scherer's equation | 0.7449 | 0.7204 | 0.6974 | 0.7211 |
| Lattice Parameter (nm) | 0.8418 | 0.8387 | 0.8382 | 0.8304 |
| Interplanary Spacing | 2.5353 | 2.5012 | 2.5258 | 2.5245 |
| X-ray Density(ρ_x) $\times 10^{-23}$ | 189.92 | 192.36 | 190.64 | 190.71 |
| Specific Surface Area (SSA) | 69.69 | 79.70 | 76.39 | 83.89 |
| Microstrain $\times 10^{-23}$ (W-H plot) | 2.73 | 2.96 | 2.58 | 3.13 |
| Dislocation Density (δ) $\times 10^{-4} (Nm^{-2})$ | 4.866 | 6.527 | 5.891 | 7.111 |

3. UV Visible Diffuse Reflectance spectroscopy(UV-DRS)

UV-Visible diffuse reflectance spectroscopy was used to examine the optical characteristics of un-doped, doped, co-doped nanoparticles. Fig.2 Shows all the samples show a large dip in the reflectance between 300 nm and 650 nm. In Fig 2, also observed that the absorption edge shift lower wavelength for the concentration of $x=0.0$ to higher wavelength $x=0.3$ concentration. The band gap value is influenced by various factors such as crystallite size, structural parameter, carrier concentrations, presence of impurities and lattice strain[21]. The diffuse reflectance (R) data is used for the determination of band gap energy by the Kubelka Munk function.

$$F(R) = \frac{(1-R)^2}{2R} \text{------(1)}$$

where, R-reflectance, F(R)-Kubelka-Munk function, A graph is plotted between $[F(R).h\nu]^2$ and $h\nu$, The band gap of $Zn_{1-x}Co_xFe_2O_4$ ($x = 0.0$ to $x = 0.3$) are found to be 3.10eV, 3.07eV, 2.98eV, and 2.92eV Respectively. The observed band gap narrowing enhances visible-light absorption, making Co-doped $ZnFe_2O_4$ nanoparticles promising candidates for applications in photocatalysis. As the un-doped and doped Co goes from ($x=0.0$ to $x=0.3$) band gap goes on decreasing, it could be due to additional energy level which is produced due to doping.

The modified Tauc function

$$F(R)h\nu = A(h\nu - E_g)^n \text{------(2)}$$

where $n = 2$ and $1/2$ represent direct and indirect transitions that give direct and indirect band gap, respectively. In general, Kubelka-Munk (KM) function F(R) given in the Equations (1) and (2) is applied to the diffuse reflectance spectra to obtain the optical band gap. [21-24]

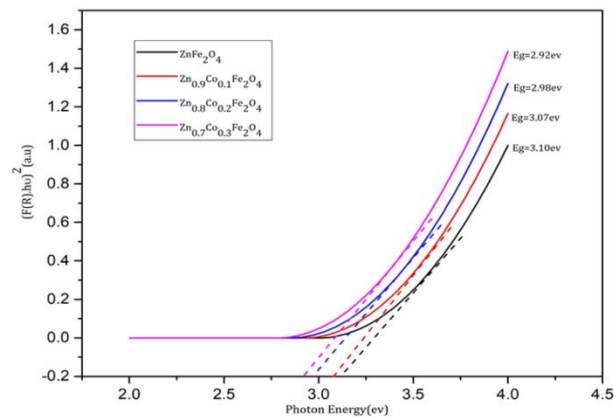


Figure.2 Tauc plots $(F(R).hv)^2$ vs. photon energy (hv) for $Zn_{1-x}Co_xFe_2O_4$ ($x = 0.0, 0.1, 0.2, 0.3$) nanoparticles. The linear extrapolation of the plots indicates progressive decrease in optical band gap energy from 3.10 eV ($x = 0.0$) to 2.92 eV ($x = 0.3$).

4. Photocatalytic Degradation.

Using the sol-gel auto-combustion method, zinc ferrite nanoparticles doped with cobalt have been synthesized and evaluated for their photocatalytic activity. Photocatalytic performance was assessed using the degradation of methylene blue dye under visible light. Using methylene blue solution dispersed in 0.15mg of the catalyst in 100 mL, the degradation percentage of the catalyst was monitored over a 60-minute period in each sample (10 min difference), in 100 mL Methylene Blue solution 0.15mg of the catalyst was dispersed. and the degradation percentage was taking up to a 60-minute period. Observed the un - doped $ZnFe_2O_4$ ($x = 0.0$) exhibited the lowest photocatalytic efficiency, reaching approximately ~ 45% degradation. As the doping of cobalt increases a significant enhancement in degradation efficiency was observed.

$Zn_{0.9}Co_{0.1}Fe_2O_4$ showed ~ 61% degradation, $Co_{0.2}Zn_{0.8}Fe_2O_4$ achieved ~ 67%, and $Co_{0.3}Zn_{0.7}Fe_2O_4$ reached a maximum of ~ 73% degradation within the same duration of time(fig.3). As per the improvement in photocatalytic performance show that the better visible light absorption, efficient charge separation, and reduced electron-hole recombination due to the incorporation of Co^{2+} ions into the Zinc ferrite structure. $Zn_{1-x}Co_xFe_2O_4$ photocatalysts under visible light respectively [25 -30]

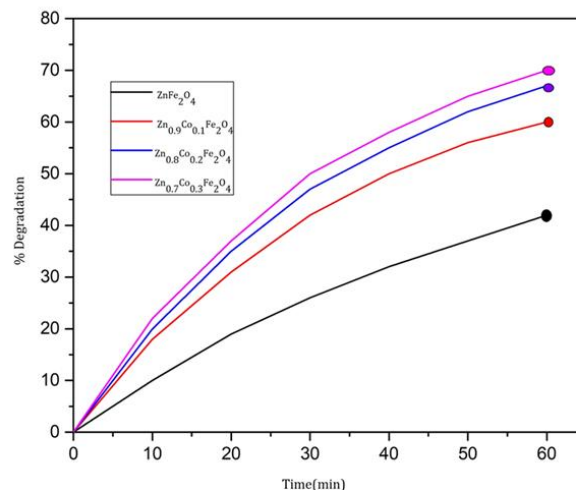


Figure.3 Time-dependent photocatalytic degradation of methylene Blue dye under visible light irradiation using $Zn_{1-x}Co_xFe_2O_4$ ($x=0.0, 0.1, 0.2, 0.3$)

Conclusion:

Sol–Gel auto combustion method is successfully applied to synthesize micro particles of cobalt-doped zinc ferrite. According to this XRD plot, cobalt ions were incorporated and zinc ferrite formed. Synthesized ferrite exhibited cubic structure with space group $Fd\bar{3}m$. According to UV Diffuse Reflectance Spectroscopy as the doping of cobalt ion increases ($x=0.0$ to $x=0.3$) band gap of material goes on decreasing. In the Photocatalytic degradation at $x=0.3$ got highest degradation $\sim 73\%$. As the band gap goes on decreasing enhancing the photocatalytic performance.

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Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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