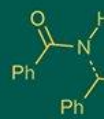


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Enhanced photocatalytic properties of La³⁺ doped zinc ferrite nanoparticles for the degradation of harmful organic dye

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Abstract

Lanthanum-doped zinc ferrite nanoparticles were prepared through the sol-gel auto-combustion technique and assessed for their photocatalytic ability in degrading methylene blue (MB) dye under visible light exposure. Various doping levels (2%, 5%, and 10%) of La³⁺ were introduced to observe their influence on the structural and optical behavior of the material. The synthesized samples were characterized using X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), UV-Vis diffuse reflectance spectroscopy (DRS), and field emission scanning electron microscopy (FESEM). XRD results confirmed the formation of a single-phase spinel structure, with minor shifts in diffraction peaks indicating the integration of La³⁺ into the lattice. FTIR spectra displayed typical metal-oxygen bonds. The UV-DRS analysis revealed a notable reduction in band gap energy as the lanthanum content increased, promoting enhanced absorption in the visible range. FE-SEM images showed morphological improvements and more uniform particle distribution due to La doping. ICP-OES data verified the chemical compositions based on different samples. Photocatalytic experiments were performed under visible light to study MB dye degradation, measured using UV-Vis spectrophotometry at $\lambda_{max} \approx 664$ nm. Among all samples, the 5% La-doped ferrite demonstrated the highest degradation efficiency. This improved activity is attributed to narrowed band gap, better light harvesting, and more effective separation of photo-generated charge carriers.

Keywords: La doping, sol-gel technique, nanoparticles, photocatalyst, methylene blue

Introduction

All living organisms, including humans, animals, and plants, depend on clean and safe water for survival. Historically, water was freely accessible, but the onset of industrialization has led to significant environmental degradation [1]. The quality of water bodies has deteriorated due to the release of pollutants from various industries such as petrochemicals, pharmaceuticals, chemicals, textiles, cosmetics, and food processing [2]. Wastewater contains a variety of contaminants, including organic compounds, inorganic metal ions, and harmful microorganisms. One major concern is synthetic dyes, which pose a serious environmental threat. Azo dyes, accounting for approximately 50% to 70% of synthetic dye pollutants, are particularly problematic in industrial wastewater [3]. These dyes are toxic and potentially carcinogenic, leading to serious environmental hazards, especially when discharged into aquatic ecosystems.

Among the various purification approaches, advanced oxidation processes (AOPs) have emerged as promising techniques for breaking down toxic organic substances using semiconductor materials that rely on generating hydroxyl radicals—highly reactive and non-selective oxidants capable of degrading a broad spectrum of organic compounds in transforming stubborn pollutants into simpler, biodegradable compounds and ultimately converting them into harmless end products like carbon dioxide and water [4]. The process involves activating a semiconductor with UV or visible light, which excites electrons from the valence band to the conduction band, creating electron-hole pairs (e^-/h^+) which then interact with pollutants on the catalyst surface, leading to their reduction and/or oxidation. The advantage of semiconductor nanoparticles lies in their narrow band gaps, which allow them to harness visible light, making them highly attractive for wastewater treatment applications.

Spinel ferrites have gained interest as visible-light-active and biocompatible photo catalysts [5]. With the general formula MFe_2O_4 , these materials offer tunable properties valuable for environmental applications [6]. Zinc ferrite ($ZnFe_2O_4$) is widely studied as a semiconductor photocatalyst but is limited by rapid charge recombination and low efficiency. To enhance performance, researchers have developed doped or composite variants. Doping modifies the electronic structure by introducing localized energy states within the band gap, which reduces the recombination of photogenerated electron-hole pairs and extends its ability to absorb visible light. It also increases the surface area and porosity of the material, providing more active sites for pollutant interaction. Doping with trivalent ions like La or Gd reduces the band gap and boosts photocatalytic activity [7, 8]. La^{3+} -doped $ZnFe_2O_4$ exhibited significantly enhanced photocatalytic degradation of rhodamine B, nearly doubling the efficiency compared to un-doped $ZnFe_2O_4$. In a normal spinel structure, M^{2+} ions predominantly occupy the tetrahedral sites, whereas Fe^{3+} ions reside in octahedral sites. In mixed spinel, both M^{2+} and Fe^{3+} ions are distributed over tetrahedral and octahedral positions. This arrangement plays a key role in determining the material's magnetic, electrical, and structural properties. In addition, zinc ferrite possesses several features that enhance its photocatalytic activity [9]. Moreover, factors such as synthesis method, reaction temperature, pH, and calcination conditions also significantly affect the characteristics of the final ferrite product.

Incorporation of La^{3+} in Zn-Mg nano ferrites and Co-Cu ferrite nanoparticles improved their magnetic, dielectric, and photocatalytic properties for dye removal [10]. Among various RE ions, lanthanum (La^{3+}) has attracted considerable interest due to its large ionic radius and strong 4f orbital interactions, which can improve charge separation and enhance surface activity [11]. It is reported [12] that nano particles of zinc-copper ferrite achieved 94% degradation efficiency. Photocatalytic efficiency of 98% has been reported [13] for Nd-doped Ni-Zn ferrite degrading methylene blue in 90 minutes. Rare-earth (RE) elements typically exist in a +3-oxidation state and possess 4f orbitals, which can play a crucial role in charge carrier dynamics by serving as trapping centres, thereby reducing the recombination rate of photogenerated electron-hole pairs. Nickel-substituted cobalt-zinc ferrite nanoparticles have reported to enhance dye degradation with higher nickel concentrations [14].

The present study focuses on the synthesis of La^{3+} -doped $ZnFe_2O_4$ nanoparticles at varying concentrations using the sol-gel method. The preparation and characterization of $ZnFe_{2-x}La_xO_4$ through this technique are detailed. The photocatalytic efficiency of the lanthanum-doped zinc ferrite is evaluated through the degradation of methylene blue in an aqueous solution which is harmful to human health above a

certain concentration due to its substantial toxicity [15].

Materials and Methods

Freshly prepared Zinc (II) nitrate, iron (III) nitrate and lanthanum (III) nitrate with required stoichiometric ratios were dissolved in water. Aqueous solution of citric acid was added in 3:1 molar ratio. It was then stirred at an initial temperature of about $120^{\circ}C$ till a viscous gel was obtained, which upon further heating (up to $200^{\circ}C$) undergoes an auto-combustion reaction resulting in rapid formation of porous ferrite powder. It was allowed to dry and ground into a fine powder. The resulting product was then calcined in a muffle furnace at $700^{\circ}C$ for 3 hours. The samples were marked as P, Q and R for the lanthanum doping percentage of 2%, 5% and 10% respectively.

An aqueous solution of methylene blue was prepared at suitable concentrations. Lanthanum-doped zinc ferrite nanoparticles were introduced into the dye solution and ultrasonicated for 20 minutes. The pH of the methylene blue solution was adjusted to an alkaline range between 10 and 12. Before initiating light exposure, the reaction mixture containing the dye and La-doped zinc ferrite nanoparticles was stirred in the dark for 30 minutes for achieving adsorption-desorption equilibrium between the methylene blue molecules and the surface of the photocatalyst, ensuring that any subsequent changes were due to photocatalytic action rather than simple adsorption. Then the mixture was irradiated using a 100-watt visible light bulb placed inside a controlled photocatalytic chamber 15 cm above the solution surface to begin the degradation process.

Results and Discussion

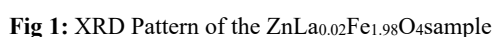
XRD Analysis

The structural and phase characterization of the newly synthesized lanthanum-doped zinc ferrite ($La_xZnFe_{2-x}O_4$) nanoparticles was performed using a Cu $K\alpha$ X-ray diffractometer (RIGAKU ULTIMA IV, $\lambda=0.15406$ nm) operating at 40 kV and 40 mA. The XRD analysis confirmed the crystalline structure of the nanoparticles. Major diffraction peaks were observed in the 2θ range of 30.071° to 36.92° . The lack of extra peaks in the XRD pattern confirms the phase purity of the synthesized samples showing that there are no secondary phases or impurities present (Fig. 1).

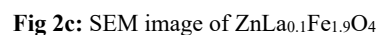
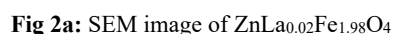
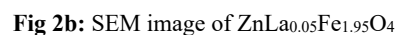
The crystallite size of $ZnLa_xFe_{2-x}O_4$ ($x = 0.02, 0.05, \text{ and } 0.1$) nanocrystals were determined using the Debye-Scherrer formula:

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

Where D is the crystallite size, K is the Scherrer constant, λ is the wavelength of the X-ray source (0.1540 nm), β is the full width at half maximum (FWHM) of the peak, and θ is the Bragg angle. The crystallite size of the NPs ($x = 0.02, 0.05, 0.1$) was found to be approximately 28, 29, and 31 nm, respectively.



A Field Emission Scanning Electron Microscope (ZEISS Gemini 300) was used to examine the morphology and structural properties of the lanthanum-doped zinc ferrite nanoparticles. The nanoparticles mostly exhibit a quasi-spherical to polyhedral morphology (Fig.2a-c) and emphasized distinct faceted crystalline areas. In contrast to the un-doped ferrites, lanthanum doping showed a marked rise in agglomeration upon increasing its doping concentration, generating densely packed clusters and porous aggregates that alter the dynamics of surface energy and crystal development. The resulting nanostructure, with its high surface area and interconnected pores, is expected to improve surface-driven interactions, facilitate ion diffusion, and potentially enhance performance in applications such as catalysis, sensors, and energy storage devices.



EDAX-Analysis

Energy Dispersive X-ray Spectroscopy is a powerful tool used in conjunction with a Scanning Electron Microscope (SEM) to analyze elemental composition of the samples. The presence of La, Zn, Fe, and O atoms causes the associated peaks in the spectrum. The observed peaks

matched well with the expected stoichiometry derived from the doping levels used in the synthesis process (Table-1). Notably, the progressive increase in La peak intensity at higher doping concentrations indicates successful incorporation of lanthanum into the ferrite lattice (Fig. 3a-c).

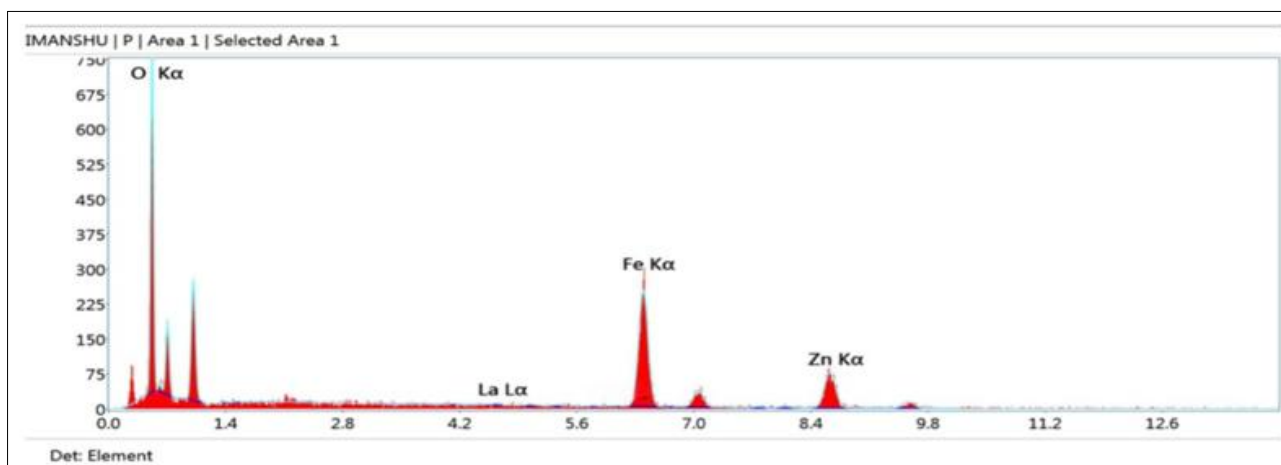


Fig 3a: EDAX patterns of synthesized $\text{ZnLa}_{0.02}\text{Fe}_{1.98}\text{O}_4$

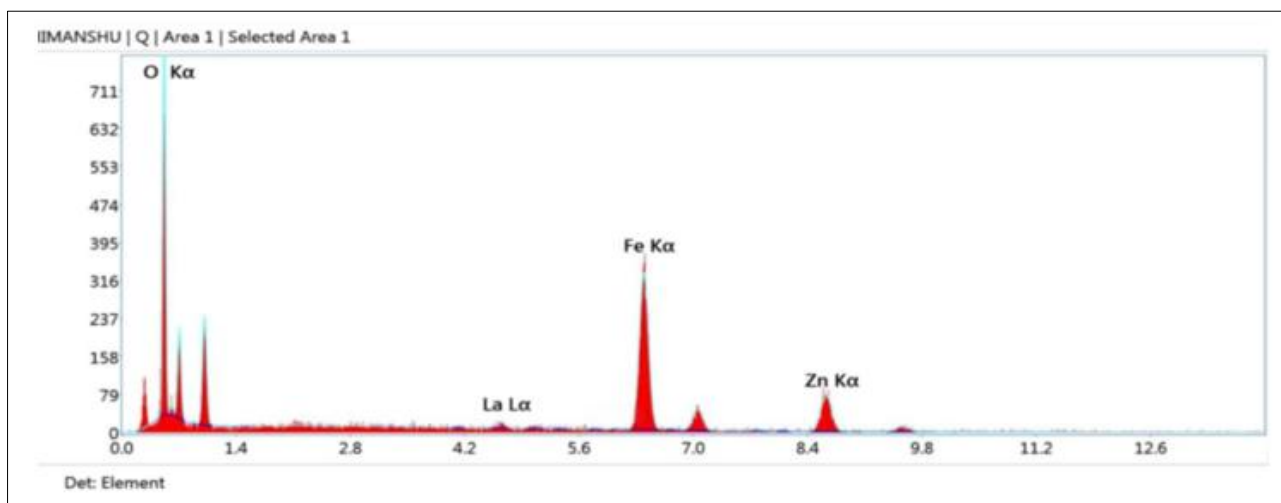


Fig 3b: EDAX patterns of synthesized $\text{ZnLa}_{0.05}\text{Fe}_{1.95}\text{O}_4$

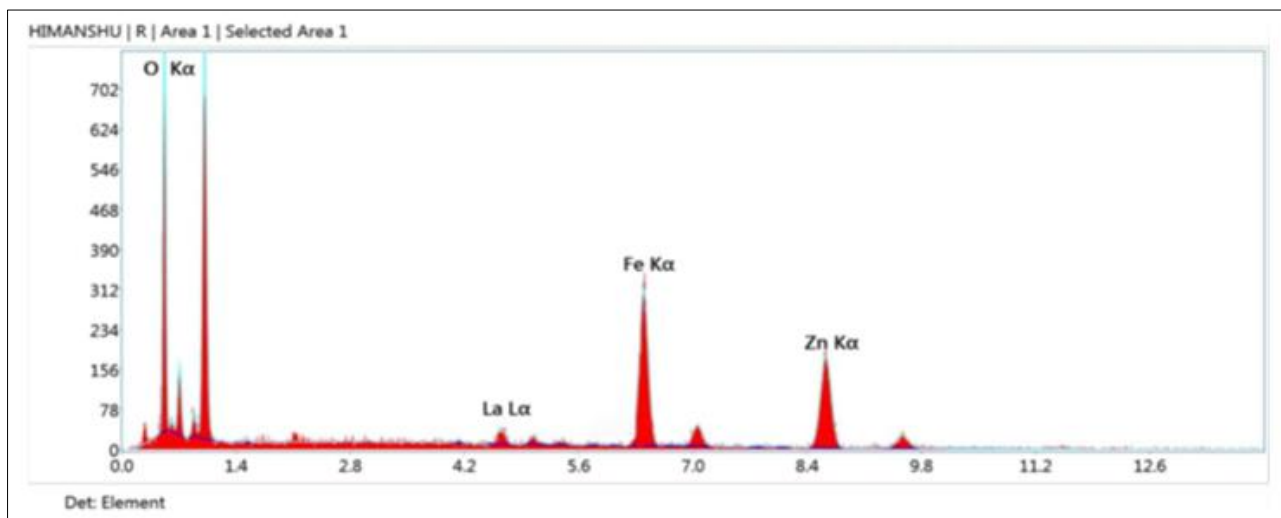


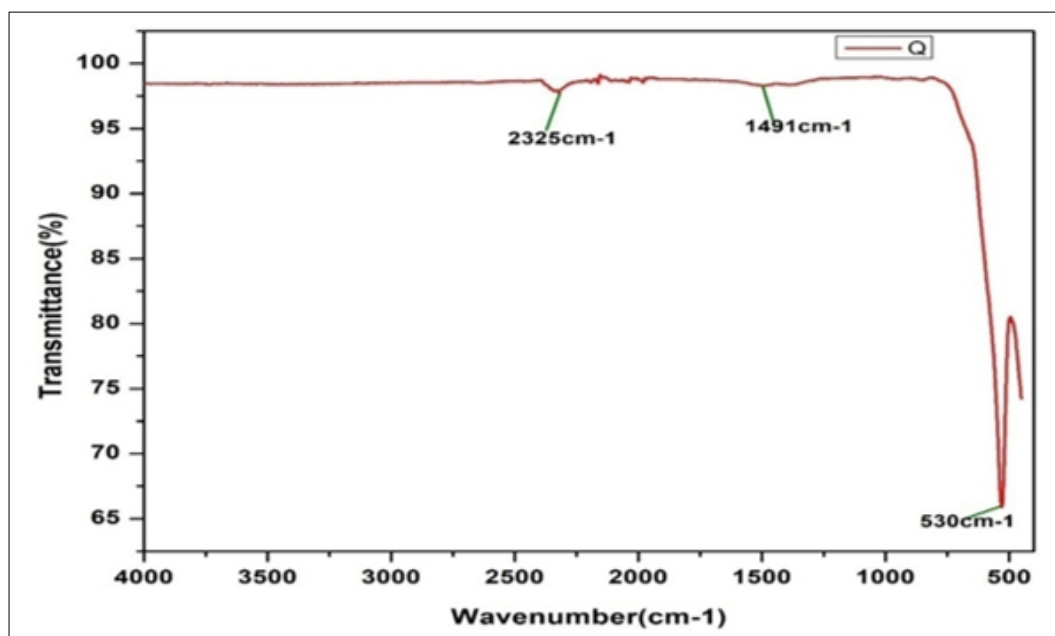
Fig 3c: EDAX patterns of synthesized $\text{ZnLa}_{0.1}\text{Fe}_{1.9}\text{O}_4$

Table 1: EDAX elemental composition of synthesized doped Zinc ferrites P, Q and R

Elements	Weight%			Atomic%		
	P	Q	R	P	Q	R
O K	36.8	33.1	28.7	68.8	65.3	61.6
Fe K	34.9	40.3	26.5	18.7	22.7	16.3
Zn K	26.9	23.5	39.9	12.3	11.3	20.9
La L	0.8	2.4	4.3	0.2	0.5	1.1

FTIR Analysis: The FTIR analysis of lanthanum-doped zinc

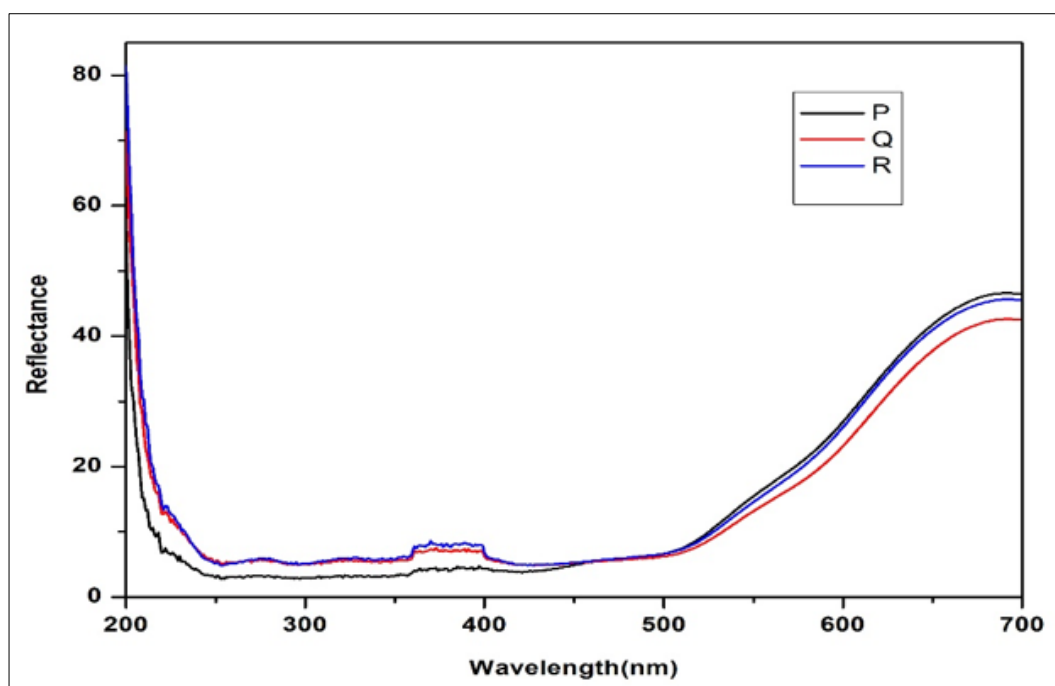
ferrite was performed using the FTIR spectrophotometer (Spectrum Tool 104789). A significant peak around 530 cm^{-1} is associated with stretching vibrations of the Fe-O bond (Fig.4), suggesting alterations in the Fe-O-Fe bond length and indicating strong interactions between the metal and oxygen atoms in the spinel lattice. The band at 1491 cm^{-1} may be linked to minor surface-adsorbed species or residual organic groups from the synthesis process. These spectral features collectively confirm the structural integrity and bonding environment within the doped ferrite material.

**Fig 4:** FTIR spectra of synthesized $\text{ZnLa}_{0.05}\text{Fe}_{1.95}\text{O}_4$

Optical Properties

UV-Visible Diffuse Reflectance Spectroscopy (UV-DRS) is widely used to investigate the optical properties and

estimate the band gap of powdered materials. Fig. 5 presents the UV-DRS results of $\text{ZnLa}_x\text{Fe}_{2-x}\text{O}_4$ samples with doping levels of $x = 0.02$ (P), 0.05 (Q), and 0.1 (R).

**Fig 5:** UV-DRS analysis of $\text{ZnLa}_x\text{Fe}_{2-x}\text{O}_4$

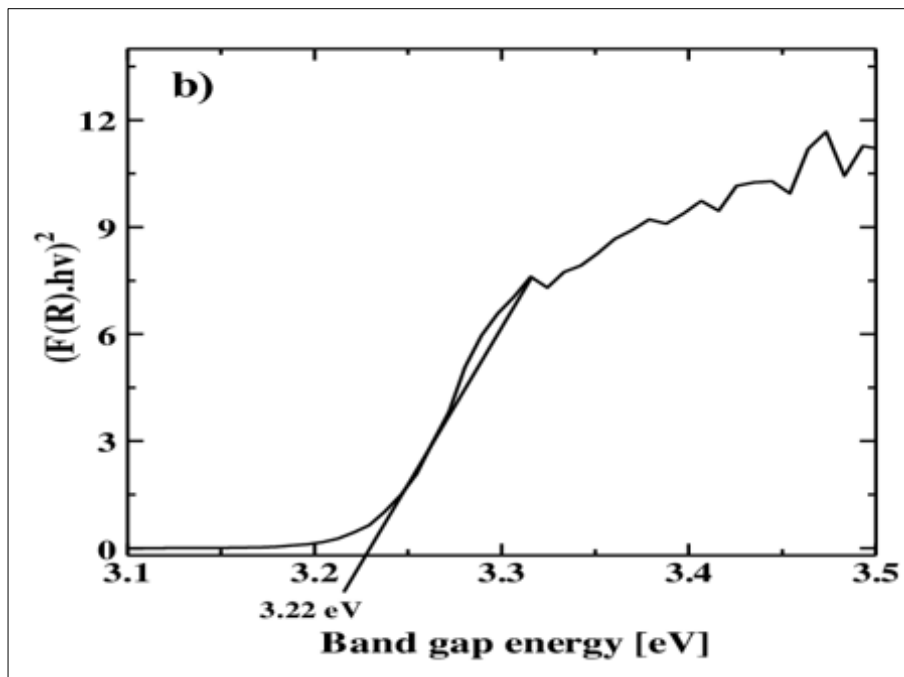


Fig 6 (a): Tauc plot for Band gap energy of zinc ferrite

A Tauc plot is then employed in order to determine the band gap energy (E_g). As seen in Fig. 6(a), the band gap for undoped zinc ferrite ZnFe_2O_4 , was found to be 3.22 eV. The substitution of larger La^{3+} ions for Fe^{3+} ions brings about lattice distortions that alter the local environment and shift electron energy levels. In the band gap, this doping also produces defect states that serve as an intermediary level that aid electron movement from the valence band to the

conduction band. Moreover, La^{3+} doping alters the distribution of Zn^{2+} , Fe^{3+} ions within the crystal, influencing the overlap of Fe 3d and O 2p orbitals. As a result of these effects, the band gap narrows to 2.27 eV at 2% La, 2.23 eV at 5% La, and 2.25 eV at 10% La (Fig.6b). These changes are especially important for enhancing photocatalytic activity in the visible spectrum.

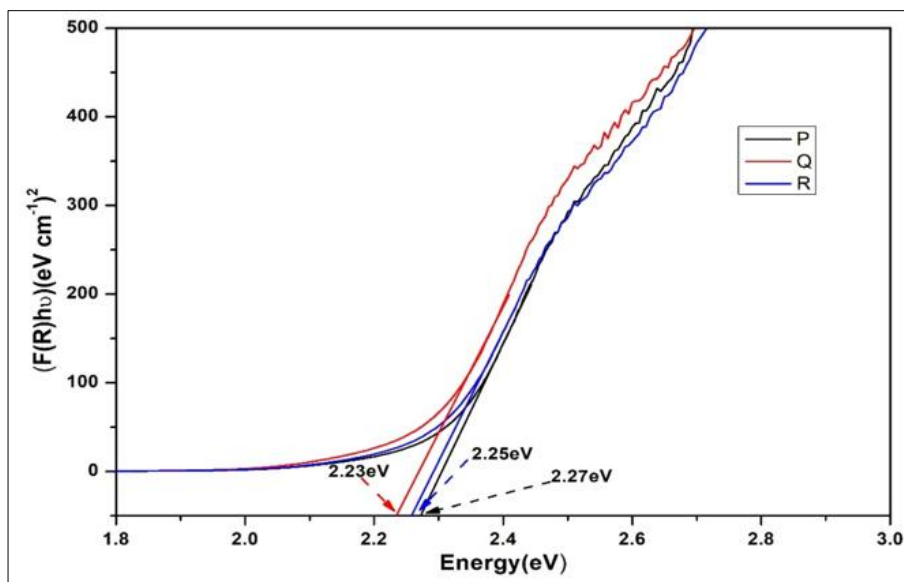


Fig 6(b): Band gaps of P($\text{ZnLa}_{0.02}\text{Fe}_{1.98}\text{O}_4$), Q($\text{ZnLa}_{0.05}\text{Fe}_{1.95}\text{O}_4$), and R($\text{ZnLa}_{0.1}\text{Fe}_{1.9}\text{O}_4$)

ICP-OES Analysis

Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) was used to determine the concentrations of Fe, Zn, and the rare-earth dopant (La). As the proportion of La increased, the Fe content decreased accordingly, confirming

the replacement of Fe^{3+} ions by trivalent dopants (Table 2). The Zn content remained nearly unchanged, suggesting that the Zn^{2+} positions in the spinel lattice were unaffected by doping. The results validate the successful substitution process by rare earth metal.

Table 2: ICP-OES data of 2%,5% and 10% La-doped zinc ferrite

Sl. No.	Sample weight	Acid used	Fe conc. (mg/L)	Fe%	Zn conc. (mg/L)	Zn%	La conc. (mg/L)	La%
P	0.0510	Aqua Regia	484.54	47.45	425.6	41.75	17.06	1.66
Q	0.0431	Aqua Regia	450.55	46.57	357.2	41.43	41.00	4.75
R	0.0504	Aqua Regia	427.49	42.19	443.15	43.96	97.27	9.64

Photocatalytic Study

The photocatalytic efficiency is mainly due to the improved separation of photogenerated electron-hole pairs and the increased surface area of the nanomaterials, allowing efficient interaction with dye molecules. The rate of dye removal varied slightly with changes in catalyst concentration, highlighting the role of catalyst dosage in optimizing photocatalytic performance. As the photocatalytic reaction proceeded, the intense blue color of methylene blue (MB) solution was progressively lightened

and eventually turned almost colorless, indicating substantial dye degradation.

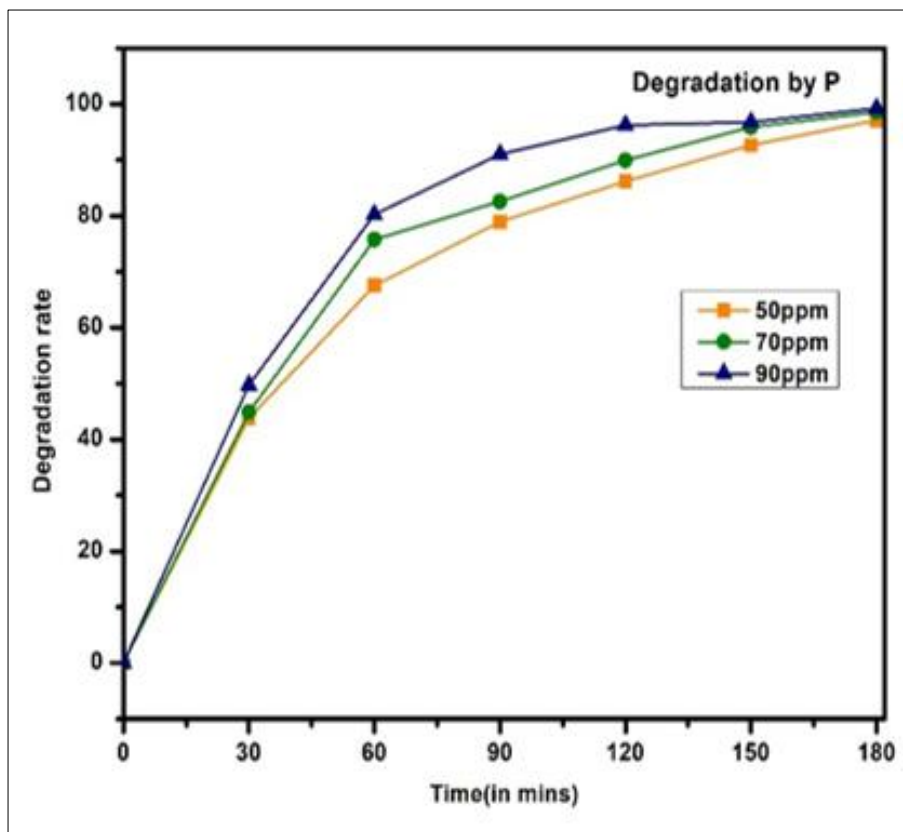
The extent of MB degradation was determined using the following equation:

$$D\% = (A_0 - A)/A_0 \times 100\% \dots\dots\dots(2)$$

Where; A_0 represents the initial absorbance of the MB solution & A is the absorbance measured at a specific time interval. Analyses were done keeping MB concentration constant while varying catalyst concentration and vice versa.

Table 3: % Degradation data of 3ppm MB dye by different catalytic concentrations of P, Q & R

Time (mins)	ZnLa _{0.02} Fe _{1.98} O ₄ (P)			ZnLa _{0.05} Fe _{1.95} O ₄ (Q)			ZnLa _{0.1} Fe _{1.90} O ₄ (R)		
	5mg/L	7mg/L	9mg/L	5mg/L	7mg/L	9mg/L	5mg/L	7mg/L	9mg/L
30	43.806	44.883	49.730	42.728	44.524	56.912	38.061	40.574	55.296
60	67.504	75.763	80.251	72.351	77.019	78.096	66.786	70.736	73.249
90	78.994	82.585	91.023	86.175	88.868	90.484	80.430	86.179	88.330
120	86.175	89.946	96.229	94.254	91.382	94.075	89.228	93.895	94.793
150	92.639	95.870	96.768	95.870	96.409	96.768	93.895	95.511	96.409
180	97.127	98.563	99.281	98.025	98.922	99.281	96.229	97.845	98.204

**Fig 7(a):** D% of MB dye by different concentrations of P

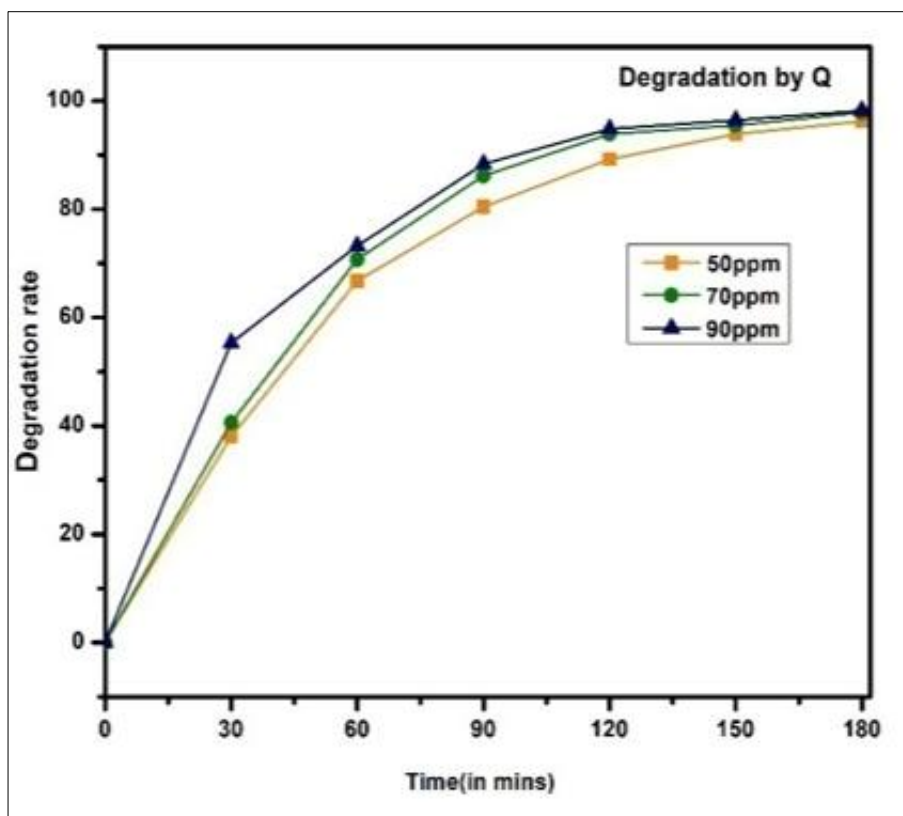


Fig 7(b): D% of MB dye by different concentrations of Q

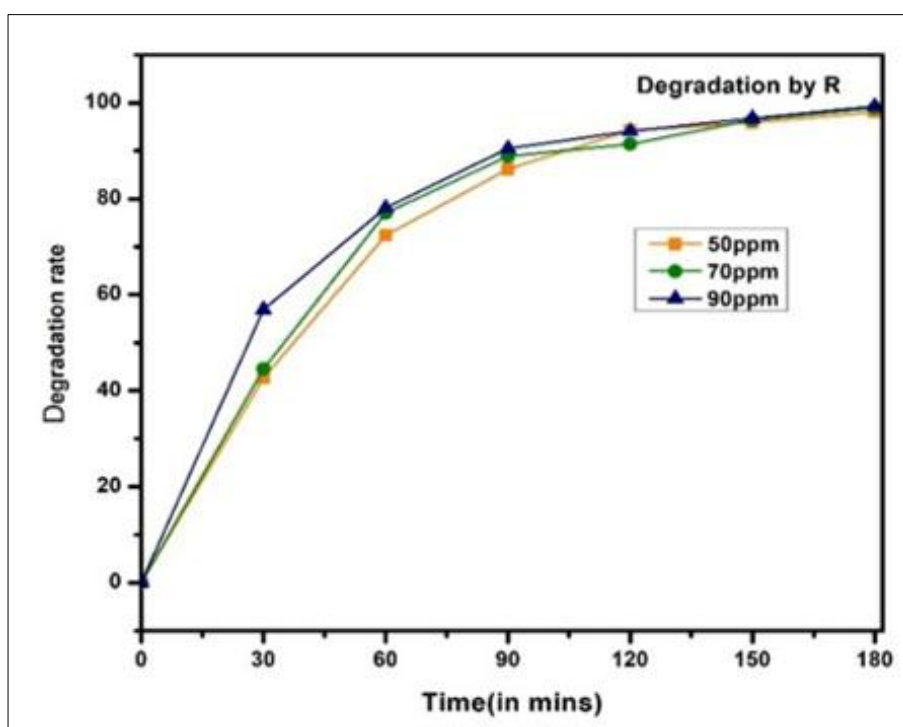


Fig 7(c): D% of MB dye by different concentrations of R

In all three plots (Fig.7a-c), the degradation rate increases with time, approaching nearly 100% degradation by 180 minutes. For all samples (P, Q, and R), the highest degradation rates are generally observed at 90 ppm concentration, followed by 70 ppm, and then 50 ppm. This indicates that increasing the catalyst concentration enhances the degradation efficiency. While all catalysts show similar trends, the degradation rates at different times appear to be slightly different. Catalyst R seems to achieve slightly

higher degradation rates earlier compared to P and Q, suggesting it might be more efficient initially.

The table-3 illustrates the photocatalytic degradation efficiency of La-doped zinc ferrite ($\text{ZnLa}_x\text{Fe}_{2-x}\text{O}_4$) at different doping levels ($x = 0.02, 0.05, 0.1$) and catalyst amounts (5 mg/l, 7 mg/l, 9 mg/l) over time. The results show that dye removal improves with increase in time with longer visible light irradiation. Notably, the sample $\text{ZnLa}_{0.05}\text{Fe}_{1.95}\text{O}_4$ at 9 mg/l achieved the highest efficiency

(~99%) after 180 minutes, highlighting the accurate effect of lanthanum doping and thereby its photocatalytic performance.

Effect of Dye Concentration

The Table-4 displays the photocatalytic degradation

efficiency (%) of methylene blue over time (30 to 180 minutes) using $\text{La}_{0.05}\text{Fe}_{1.95}\text{O}_4$ catalyst at varying dye concentrations (3 ppm, 5 ppm, and 7 ppm). The degradation increases steadily with time for all concentrations. Lower concentrations (3ppm) show quicker initial degradation, likely due to better light absorption and catalyst activity.

Table 4: % Degradation data of 3ppm, 5ppm & 7ppm MB dye by Q (7 mg) catalyst

Time (in mins)	$\text{ZnLa}_{0.05}\text{Fe}_{1.95}\text{O}_4$		
	3ppm	5ppm	7ppm
30	60.795	57.298	52.426
60	68.673	62.372	60.851
90	74.875	71.921	71.718
120	87.661	85.338	82.607
150	92.923	91.217	90.373
180	99.264	97.893	96.791

The Figure-8 illustrates the degradation behaviour of methylene blue over a period of 180 minutes using $\text{La}_{0.05}\text{Fe}_{1.95}\text{O}_4$ as the photocatalyst at three different dye concentrations i.e. 3 ppm, 5 ppm, and 7 ppm. A consistent increase in degradation rate is observed with time across all concentrations, indicating effective photocatalytic activity. Concentrations 3 ppm shows a relatively faster degradation

rate in the initial stages, which is attributed to improved light penetration and more efficient interaction between dye molecules and the catalyst. As time progresses, all the dye concentrations approach near- complete degradation, reaching around 99% by the end of 180 minutes. This suggests that the catalyst remains highly efficient across varying dye concentrations.

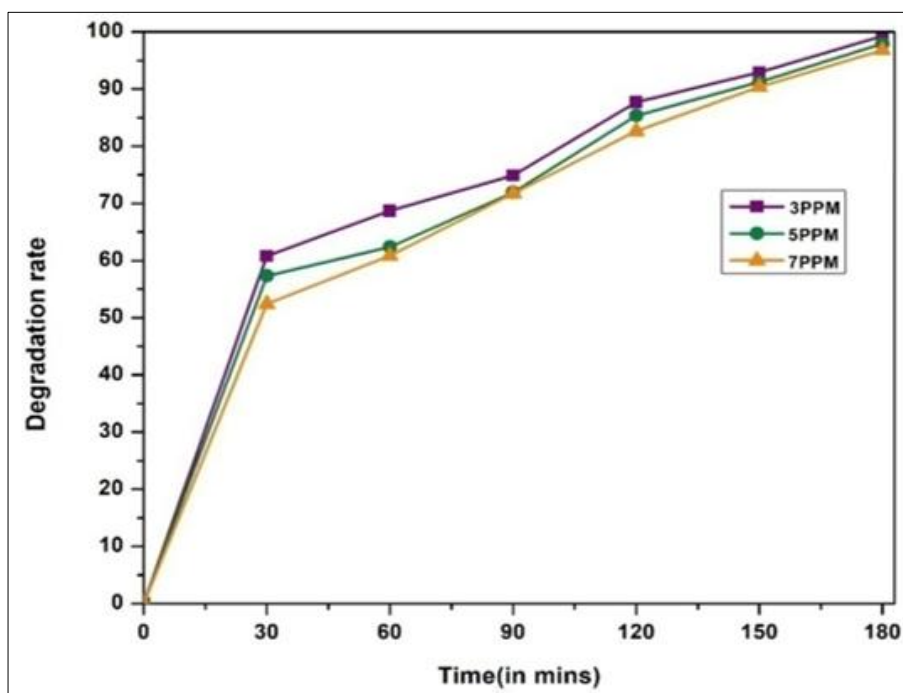


Fig 8: % Degradation of different concentrations of MB dye by Q (7mg)

Role played by the doped oxide

Lanthanum (La^{3+}) doping in zinc ferrite (ZnFe_2O_4) introduces notable changes in both structure and functionality [16, 17] due to the considerable size difference between La^{3+} (1.06 Å) and Fe^{3+} (0.645 Å). When La^{3+} replaces Fe^{3+} at the octahedral B-sites, it creates internal lattice strain, leading to distortion and unit cell expansion. This structural change is evidenced by a shift in XRD peaks to lower angles. The substitution can also result in the formation of oxygen vacancies to compensate for charge imbalance, which may improve photocatalytic efficiency. Additionally, La^{3+} incorporation can cause a redistribution of cations between tetrahedral and octahedral sites, partially inverting the spinel structure. These adjustments can lead to

band gap narrowing, which enhances visible light absorption and increases photocatalytic potential. Furthermore, La^{3+} can function as an electron trap, reducing the recombination rate of charge carriers and enhancing redox reaction efficiency. It may also improve surface area and increase the number of active catalytic sites, aiding pollutant adsorption and surface activity. Altogether, these modifications contribute to improved photocatalytic degradation performance in methylene blue dye removal.

Conclusion

The work demonstrated the successful synthesis of lanthanum-doped zinc ferrite ($\text{La-ZnFe}_2\text{O}_4$) nanoparticles with different lanthanum concentrations indicating the

effective incorporation of La^{3+} into the ZnFe_2O_4 lattice. There is a noticeable narrowing of the band gap for the 5% La-doped sample thereby improving visible light absorption and enabling efficient photocatalysis and achieved nearly 91% degradation of methylene blue within 120 minutes, compared to 60% by the undoped sample. This enhanced activity is attributed to the improved charge separation, reduced recombination of photogenerated electron-hole pairs, and better utilization of the visible spectrum. Among all the compositions, the sample with 5% La content exhibited the best balance between crystallinity, optical response, and photocatalytic efficiency. These findings demonstrate that La- ZnFe_2O_4 is a promising material for applications in wastewater treatment, particularly for the removal of organic dyes. Future investigations can delve into precise control of dopant concentration, crystallite size, and surface area, all of which directly affect the photocatalytic response.

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