

Green synthesis of Cd and Cd/Zr-dual doped CeO₂ nanoparticles for enhanced photocatalysis and latent fingerprint applications

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Green synthesis of Cd and Cd/Zr-dual doped CeO₂ nanoparticles for enhanced photocatalysis and latent fingerprint applications.

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Abstract

Cd-doped and Zr/Cd co-doped CeO₂ nanomaterials were synthesized using an eco-friendly solution combustion method with *Lobularia maritima* seed extract acting as a green fuel and reducing agent. The structural, morphological, elemental, and optical characteristics of the synthesized nanoparticles were thoroughly investigated using p-XRD, FT-IR, FE-SEM, EDX, UV-DRS, and PL spectroscopy. UV-DRS results indicated a systematic narrowing of the band gap with increasing co-doping (3.14 eV for Cd-CeO₂, 3.02 eV for Zr_{0.05}-Cd_{0.05}-CeO₂, and 2.88 eV for Zr_{0.1}-Cd_{0.1}-CeO₂), confirming enhanced visible-light absorption due to dopant incorporation. p-XRD analysis revealed a reduction in crystallite size from ~23.4 nm to ~21 nm, suggesting that co-doping effectively influences crystal growth. Among the samples, Zr_{0.05}-Cd_{0.05}-CeO₂ demonstrated the highest photocatalytic efficiency, achieving ~90% degradation of Rose Bengal under UV irradiation within 180 min through reactive oxygen species. Phytotoxicity studies verified the treated water's environmental safety, while successful latent fingerprint visualization on rigid surfaces highlighted the nanoparticles multifunctional potential in environmental remediation and forensic science.

Keywords: nanoparticles, photodegradation, scavengers, fingerprints.

1. Introduction

Conventional synthesis techniques typically involve toxic chemicals, lengthy processing times, and elevated production costs, making them less sustainable and economically viable. Green chemistry addresses these limitations by utilizing cost-effective and environmentally benign alternatives. Plant extracts, which are generous in naturally occurring functional groups such as carboxyl and hydroxyl moieties, serve as effective and sustainable substitutes for traditional chemical reagents in eco-friendly synthesis processes [1,2]. A rapidly evolving paradigm in green nanomaterial synthesis emphasizes the development of environmentally benign methodologies that modify precise control over particle morphology and surface functionality, thereby enhancing physicochemical stability. These advancements are strategically aligned with goals of environmental remediation, regulatory compliance, and the integration of sustainable practices in advanced material design and industrial process optimization [3,4,5].

Researchers have focused a lot of attention on metal oxides because of their exceptional catalytic performance and simple manufacturing techniques. Among them, CeO_2 has been significantly studied and is considered one of the most promising rare earth oxides [6,7]. Rare earth elements exhibit distinct chemical behavior compared to main group and transition metals due to the deeply embedded 4f orbitals, which are effectively shielded by outer 4d and 5p electrons. This unique electronic structure gives rise to exceptional magnetic, electronic, and catalytic properties that open up innovative possibilities in areas where

conventional metals fall short enabling next-generation technologies in energy, data storage, and advanced manufacturing [8,9].

Water pollution caused by synthetic dyes has become a major global concern and holds a significant position in environmental protection strategies and future sustainability plans [10]. The growing presence of these dyes in aquatic systems leads to long-lasting damage to marine ecosystems and poses indirect risks to other organisms, including humans, who rely on these water sources within the ecological cycle [11,12]. CeO_2 has limited visible light absorption and inefficient electron-hole pair separation during photocatalysis due to its wide bandgap. To address this limitation, it is commonly doped with transition and rare earth metals such as Co, Cr, Nb, Zr, Sm, and La. These dopants enhance photocatalytic efficiency by improving charge carrier separation and thereby promoting surface reactions [13,14,15].

Dana Toloman et al. investigated the effects of incorporating aliovalent cations such as Eu^{3+} , Gd^{3+} , and Y^{3+} into the ceria lattice, focusing on the relationships between structural and morphological characteristics, luminescence properties, and photocatalytic activity. Their study demonstrated that the enhanced photocatalytic performance is due to an increase in charge carrier concentration and light absorption capacity, resulting from defect formation induced by the dopants in the ceria lattice [16,17].

Latent fingerprints are often invisible to the naked eye and require special techniques for visualization. One cutting-edge method involves using nanoparticles to enhance the detection and visualization of latent fingerprints. This technique leverages the specific physical and chemical properties of nanoparticles such as their small size, high surface area, and ability to bind to fingerprint residues or substrates [18,19]. Applications of latent fingerprint identification using nanotechnology are becoming increasingly fascinating. Nanoparticles improve both the general features and the finer details of fingerprints, such as pore patterns and subtle

minutiae. Nanomaterials are used to develop latent prints because they significantly enhance their visibility. By improving surface-level interactions with the natural substances present on the ridges of fingerprints, they help better distinguish between individual fingerprint patterns [20]. Fingerprints are impressions formed by the inner layer of skin found on the fingers, known as friction ridge skin. This specialized skin, characterized by tiny ridges and grooves, appears on areas like the fingertips, palms, and soles of the feet. The patterns are unique to every individual and remain unchanged throughout a person's life. These ridge patterns serve as reliable identifiers and are often crucial pieces of evidence at crime scenes [21,22,23].

This research investigates, for the first time, the use of *Lobularia maritima* seed extract as a biofuel in the green synthesis of cadmium (Cd) and zirconium-doped cadmium (Zr/Cd) variants, via a solution combustion method. The resulting nanoparticles were calcined to ensure regular particle size and morphology. Extensive characterization was performed using various analytical techniques, including powder X-ray diffraction (p-XRD), scanning electron microscopy (SEM), energy-dispersive X-ray analysis (EDAX), Fourier-transform infrared spectroscopy (FT-IR), ultraviolet-visible diffuse reflectance spectroscopy (UV-DRS), and photoluminescence (PL) spectroscopy. The photocatalytic performance of the nanoparticles was evaluated through the degradation of Rose Bengal (RB) dye under UV light irradiation. To gain insights into the degradation mechanism, scavenger studies were conducted. Additionally, the recyclability of the catalysts was examined to assess their stability and reusability. The treated (degraded) water was further tested for its potential in supporting the growth of fenugreek plants. Moreover, the synthesized nanoparticles were explored for forensic applications, particularly in fingerprint detection, to assess their practical utility in criminal investigations.

2. Materials and Methods

2.1. Materials

Cerium nitrate hexa hydrate $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, cadmium nitrate hydrate $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ and Zirconyl nitrate $\text{ZrO}(\text{NO}_3)_2$ all of analytical grade chemicals were purchased from spectrochem. The seeds of *lobularia maritima* was commercially purchased and the seeds extraction was prepared by using distilled water and the prepared seed extract used as fuel.

2.2. Synthesis of Cd doped CeO_2 nanoparticles

Cerium nitrate hexahydrate (99.99% Aldrich) and cadmium dinitrate hydrate (99.99%) were employed as precursors. Each nitrate was dissolved in distilled water separately, and both solutions were mixed with 95 and 5 mol% of cerium nitrate and cadmium nitrate respectively. To the above solution of nitrate added plant seed extract (10ml) serves as a fuel and the mixture was stirred for 30 minutes to get homogeneous solution. The combustion reaction was performed at 500 °C for 5 hours in combustion chamber and finally got yellow solid and it was transferred into another crucible and performed calcination at 500 °C for 2 hours.

2.3. Synthesis of Cd/Zr dual doped CeO_2 nanoparticles

Cerium nitrate hexahydrate (99.99% Aldrich) Zirconyl dinitrate hydrate (99.9%), Cadmium nitrate hydrate (99.99% Aldrich) was employed as precursors. Each nitrate was dissolved in distilled water separately, and the solutions containing 90, 5 and 5 mol% of cerium nitrate and zirconyl nitrate and strontium nitrate respectively and are mixed. To the above solutions of nitrate added plant seed extract (10ml) serves as a fuel and the mixture was stirred for 30 minutes to get homogeneous solution. The combustion reaction was performed at 500 °C for 5 hours in combustion chamber and finally got yellow solid and it was transferred into another crucible and performed calcination at 500 °C for 2 hours. The synthesized nanoparticles were characterized using FT-IR, p-XRD, UV-DRS, SEM and EDX. The synthesis process of the nanoparticles is illustrated in fig.1.

3. Characterization

The crystalline nature of nanocomposites were analyzed using a Shimadzu X-ray diffractometer (PXRD-7000), employing Cu-K α radiation ($\lambda = 1.541 \text{ \AA}$). The band gap of nanoparticles was determined by Lamda-35 spectrophotometer (Perkin Elmer) operating in the wavelength range of 200-800 nm. The morphology of nanomaterials was examined via FE-SEM Hitachi-7000 spectroscopy. FT-IR spectra were obtained using a Bruker Alpha-T FT-IR spectrometer with spectral resolution of 0.7 cm^{-1} , 35 scans, and a wavenumber ($\bar{\nu}$) range from 4000 cm^{-1} to 400 cm^{-1} .

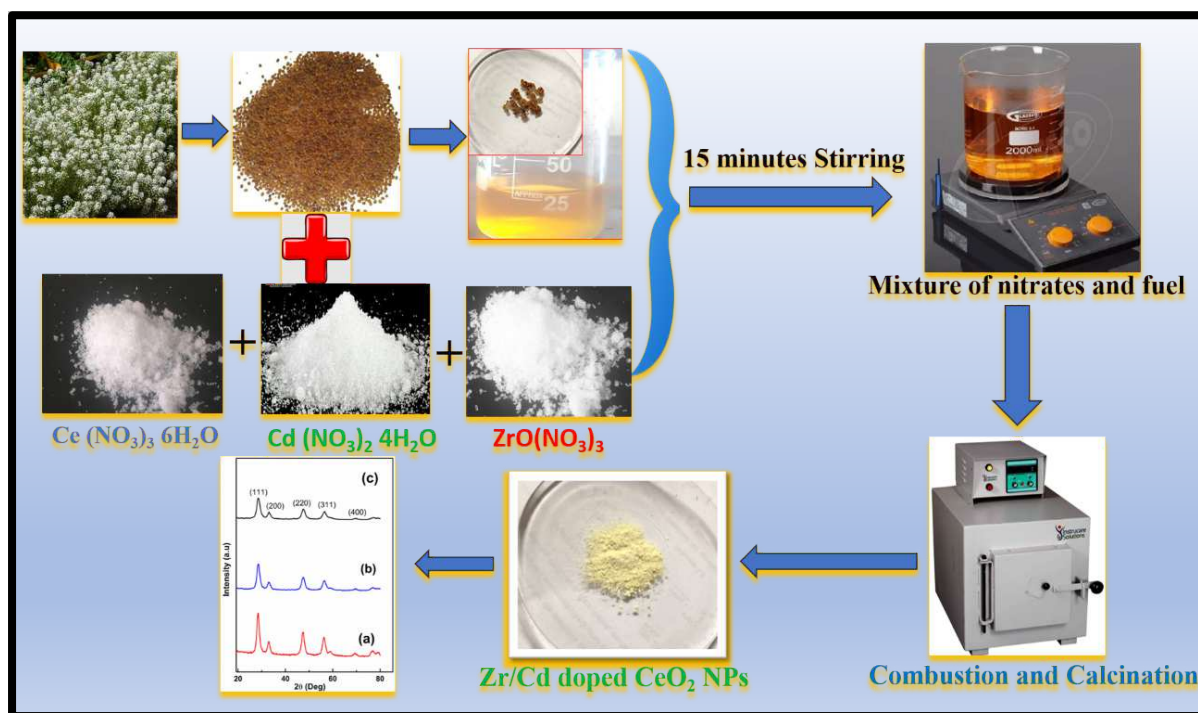


Figure 1. Graphical representation of synthesis of Cd and Zr/Cd dual doped CeO₂ nanoparticles.

3.1 Photocatalytic degradation of dye

Rose Bengal (RB) dye was used as a model pollutant to evaluate the photocatalytic performance of Cd-CeO₂, Zr_{0.05}-Cd_{0.05}-CeO₂ and Zr_{0.1}-Cd_{0.1}-CeO₂ nanoparticles under UV light irradiation using a HEBER photo-reactor. The reactor was illuminated with a Mercury-Xenon lamp (Hamamatsu L8288, 6.3 mW/cm^2), positioned 15 cm above the solution surface.. The experiments were carried out using glass trough of dimension $150 \times 75 \text{ mm}$. For each experiment, 10 mg of the synthesized nanoparticles was added to 100 mL of a 5 ppm RB dye solution. The suspension was stirred in the dark for 20 minutes to establish adsorption-

desorption equilibrium between the dye and the nanoparticles before light exposure. During UV light irradiation, 2–3 mL of the solution was sampled every 30 minutes, then centrifuged to remove catalyst particles. The absorbance of the clear supernatant was measured at 663 nm to monitor dye degradation. To study the effect of different parameters on photocatalytic efficiency, additional experiments were carried out by varying the dye concentration, catalyst dosage, and pH of the solution. The percentage degradation of dye was calculated by using following formula.

$$\% \text{ Degradation} = \frac{C_i - C_f}{C_i} \times 100 \quad \text{----- (1)}$$

Where C_i and C_f are the initial and final concentrations of dye.

3.2. Preparation of imaging LFPs by using nanoparticles

Various nonporous surfaces, including a mobile display, knife, glass slide, and mobile display, were collected from our laboratory. Latent fingerprints (LFPs) were then deposited by a single donor after washing hands with soapy water. The prepared nanoparticles were subsequently crushed using a marble pestle and mortar and gently applied to the LFPs with a soft feather brush until the fingerprints became visible. The developed fingerprints were photographed using a Samsung Galaxy-S6 tablet under both normal lighting and 365 nm UV illumination.

4. Results and discussion

4.1. P-XRD Studies

Powder X-ray diffraction (p-XRD) analysis was used to structurally characterize Cd-doped CeO_2 and Cd/Zr-dual doped CeO_2 nanoparticles. The diffraction peaks of all samples were indexed to their respective crystal planes, and their positions revealed well with the standard diffraction pattern of CeO_2 (JCPDS No. 81-0792) as depicted in fig.2(a-c) [24]. This affirms that the prepared nanoparticles exhibit a cubic fluorite crystalline structure. Importantly, the XRD pattern of the dual-doped (Cd/Zr) CeO_2 nanoparticles did not show any additional peaks, indicating that the crystal structure remains unaltered upon co-doping

[25]. This stability can be accredited to the ionic radii compatibility: Cd^{2+} (0.097 nm) closely resembles Ce^{4+} (0.097 nm), while Zr^{4+} (0.090 nm) is slightly smaller, thus allowing substitution without significant lattice distortion. A noticeable broadening of diffraction peaks and a slight shift in the 2θ values towards higher angles were noticed in the co-doped samples compared to the Cd-only doped CeO_2 . This shift is associated with a decrease in crystallite size, which is further supported by the calculated crystallite sizes using the Scherrer equation. The average crystallite sizes for $\text{Cd}_{0.05}\text{-CeO}_2$, $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ and $\text{Zr}_{0.1}\text{-Cd}_{0.1}\text{-CeO}_2$ (co-doped) nanoparticles were estimated to be 23.4 nm, 21 nm, and 18 nm, respectively. The average crystallite size and d spacing of the nanomaterials were measured using the Scherrer's equation as shown in below (Eq. (2))

$$D = \frac{K\lambda}{\beta \cos \theta} \quad \text{-----}$$

(2)

Where D is the particle size, k stands for shape factor, λ indicates the wavelength of X-ray, β is the full width of half maximum (FWHM) and θ is peak position

Lattice strain values (presented in Table 1) were calculated using the Williamson-Hall (W-H) analysis, depicted in fig. 2 (d-f). The linear slope from the W-H plot for the co-doped CeO_2 sample was steeper than that of the singly doped counterpart, suggesting increased degree of lattice strain in the Zr/Cd dual doped CeO_2 structure [26]. Importantly, the crystallite sizes estimated via the Scherrer formula differed markedly from those determined through the W-H method, which incorporates the effects of both crystallite size and microstrain on peak broadening. The modified Scherrer equation (Eq. (3)) was applied in the W-H analysis to simultaneously estimate the crystallite size ($t_{\text{W-H}}$) and lattice strain (ϵ).

$$\beta_{hkl} \cos \theta = \frac{0.9\lambda}{t_{\text{W-H}}} + 4\epsilon \sin \theta \quad \text{-----}$$

(3)

Table 1. Particle sizes and strain of Cd doped CeO₂ and Zr/Cd dual doped CeO₂ nanoparticles.

Samples	Particle sizes		Micro Strain ($\epsilon \times 10^{-3}$)
	By Scherrer's formula (nm)	By W-H Plots (t_{W-H} nm)	
Cd _{0.05} -CeO ₂	23.4	6.76	2.4
Zr _{0.05} -Cd _{0.05} - CeO ₂	21	6.05	2.78
Zr _{0.1} -Cd _{0.1} - CeO ₂	18.2	6.44	9.8

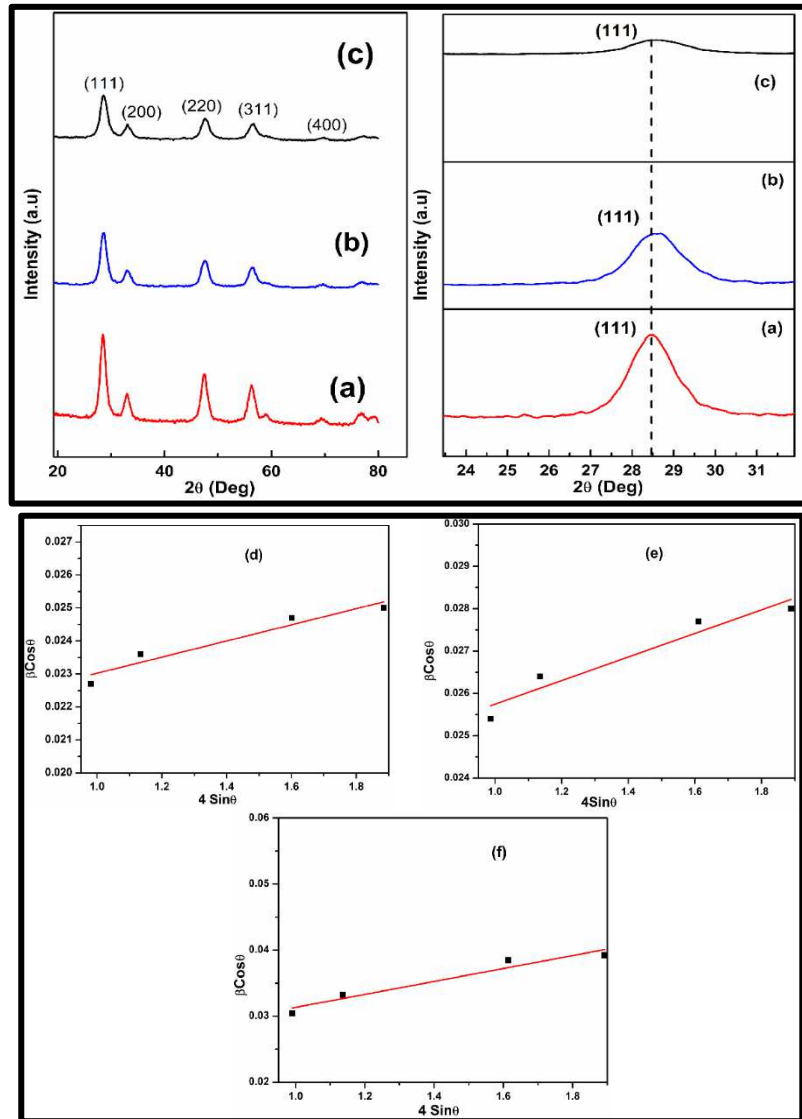


Figure 2. (a-c) p-XRD and (d-f) W-H Plots of $\text{Cd}_{0.05}\text{-CeO}_2$, $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ and $\text{Zr}_{0.1}\text{-Cd}_{0.1}\text{-CeO}_2$ nanoparticles respectively.

4.2. FT-IR analysis

The synthesized nanoparticles were characterized by using FT-IR spectroscopy in the range of $400\text{--}4000\text{ cm}^{-1}$ to investigate their vibrational characteristics as depicted in fig.3. FT-IR analysis was employed to verify the existence of metal-oxygen bonds in the metal oxide nanoparticles, as well as to determine the functional groups present. A vibrational band observed between 530 cm^{-1} and 550 cm^{-1} corresponds to the Ce-O stretching vibration [27]. Additional peaks in the range of $1050\text{--}1630\text{ cm}^{-1}$ are also attributed to Ce-O stretching vibrations, which are linked to water molecules adsorbed on the ceria surface. Due to the high surface-to-volume ratio of nanomaterials, water molecules from the surrounding environment are typically adsorbed onto their surface [28]. Furthermore, the absorption peak centred around 3422 cm^{-1} is associated with the O-H stretching vibrations of water molecules [29].

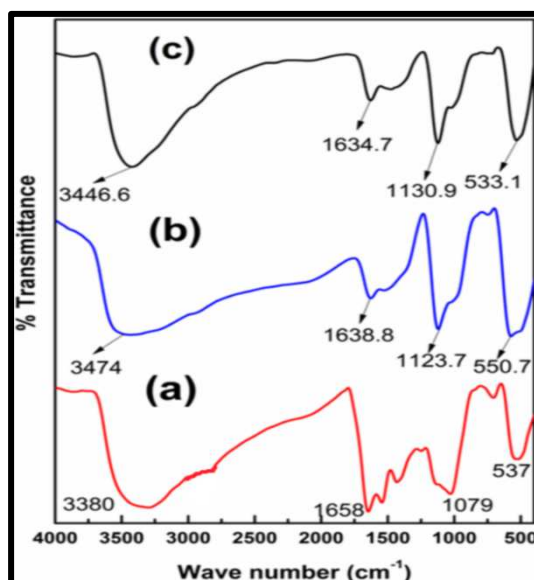


Figure 3. FT-IR spectra of $\text{Cd}_{0.05}\text{-CeO}_2$ (a), $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ (b) and $\text{Zr}_{0.1}\text{-Cd}_{0.1}\text{-CeO}_2$ (c) NPs.

4.3. SEM-EDX analysis

The morphological characteristics of Cd-doped and Cd/Zr co-doped nanomaterials were examined via scanning electron microscopy (SEM), as depicted in fig.4(a-c). The low-magnification SEM images revealed that the synthesized catalyst exhibited an irregular dispersion with distinctly separated layers and flake-like morphologies of variable thickness [30]. This heterogeneous structural organization contributed to a highly porous framework, indicative of mesoporous characteristics. The evident porosity determined in the micrographs suggests the existence of interconnected channels favorable for catalytic activity [31]. The formation of this ordered architecture is attributed to the precisely controlled synthesis parameters employed during the catalyst preparation process.

The elemental composition of the synthesized nanomaterial was analyzed using energy-dispersive X-ray spectroscopy (EDX). The EDX results affirm the existence of the constituent elements Ce, Cd, Zr, and O, along with trace amounts of N and C, which are likely residues from the XPS analysis process (fig.4d-e). The EDX spectrum demonstrated a high degree of purity, with no detectable impurity-related peaks [32]. Furthermore, the elemental stoichiometry derived from EDX elemental mapping exhibited strong agreement with the theoretical values predicted from the synthesis protocol.

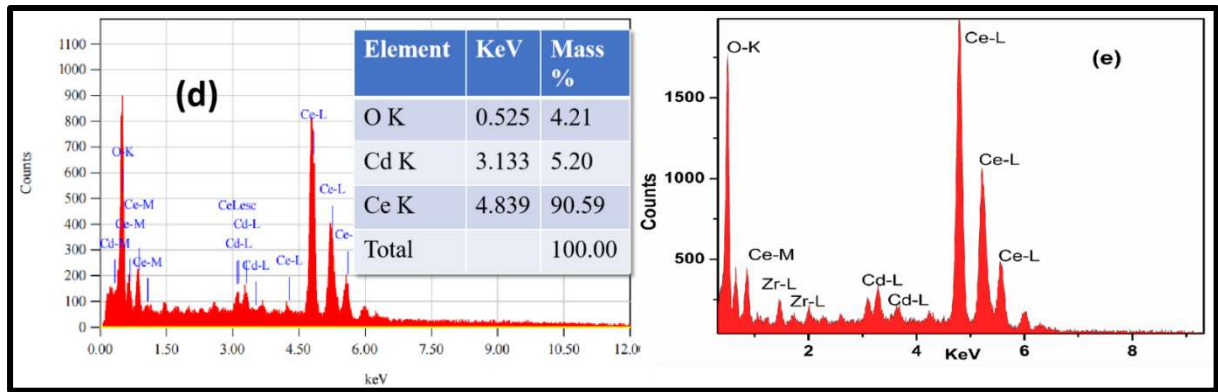
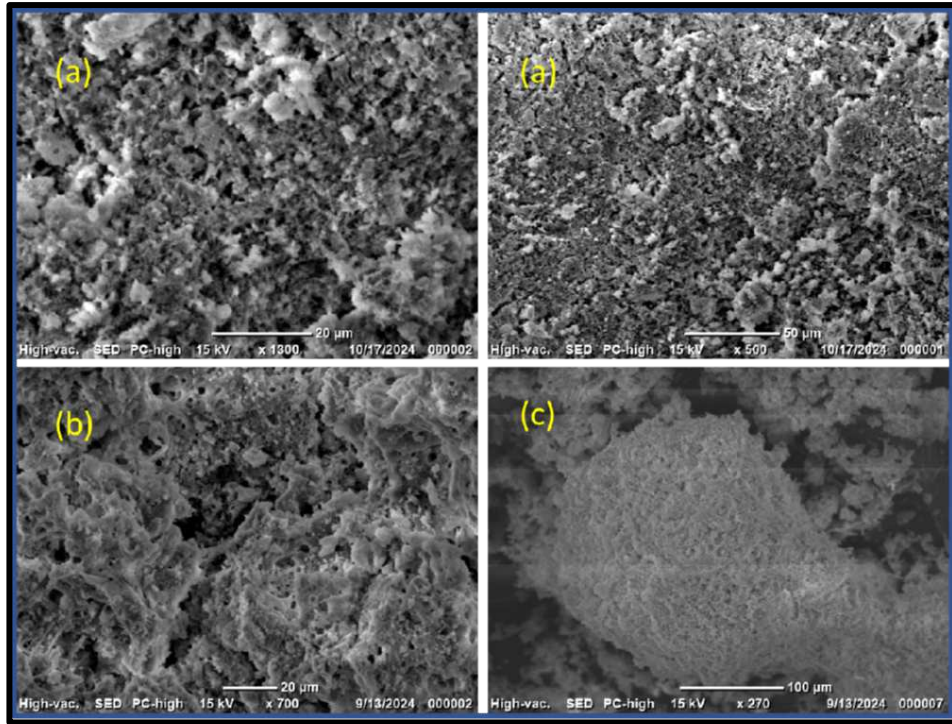


Figure 4. SEM images of $\text{Cd}_{0.05}\text{-CeO}_2$ (a), $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ (b) $\text{Zr}_{0.1}\text{-Cd}_{0.1}\text{-CeO}_2$ (c), and EDX Spectra of $\text{Cd}_{0.05}\text{-CeO}_2$ (d), $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ (e) NPs.

4.4. UV-DRS analysis

UV-DRS spectroscopy was employed to examine the optical characteristics of the nanoparticles within the wavelength range of 400–900 nm. The UV-visible absorbance spectra for Cd/CeO₂ and Cd/Zr co-doped nanoparticles are presented in fig.5(a-c). As pointed out in this figure, the absorbance peak appears below 400 nm, which is agreement with findings reported in previous literature [33]. To determine the energy gap of the synthesized nanoparticles, the Kubelka-Munk function was utilized to transform the UV-vis diffuse reflectance data into Tauc plots, as illustrated in the fig.5(d-

f). The correlation between the band gap energy and the diffuse reflectance spectrum is described by Equation (4).

$$(F(R)h\nu)^{1/\gamma} = B (h\nu - E_g) \dots \dots \dots (4)$$

In this equation, h represents Planck's constant, ν is the photon frequency, E_g denotes the band gap energy, and B is a constant. The exponent factor γ relies on the type of electronic transition in the nanoparticles. Since CeO_2 exhibits an indirect electronic transition, γ is taken as 2, and thus Equation (4) is modified accordingly [34].

$$(F(R)h\nu)^{1/2} = B (h\nu - E_g) \dots \dots \dots (5)$$

As described by Equation (5), the band gap energy is determined by plotting the y-axis as $(F(R) \cdot h\nu)^{1/2}$ and the x-axis as $h\nu$. The band gap energy (E_g) is identified at the point where the extrapolated straight-line portion of the curve intersects the x-axis. The sequence of the band gap values for the nanoparticles is as follows: $\text{Cd}_{0.05}\text{-CeO}_2$ (3.23 eV) > $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ (3.0 eV) > $\text{Zr}_{0.1}\text{-Cd}_{0.1}\text{-CeO}_2$ (2.92 eV). The observed reduction in band gap energy can be attributed to the replacement of Ce^{4+} ions with Cd^{2+} and Zr^{2+} ions, which reduces the energy difference between the O (2p) and Ce (4f) orbitals [35].

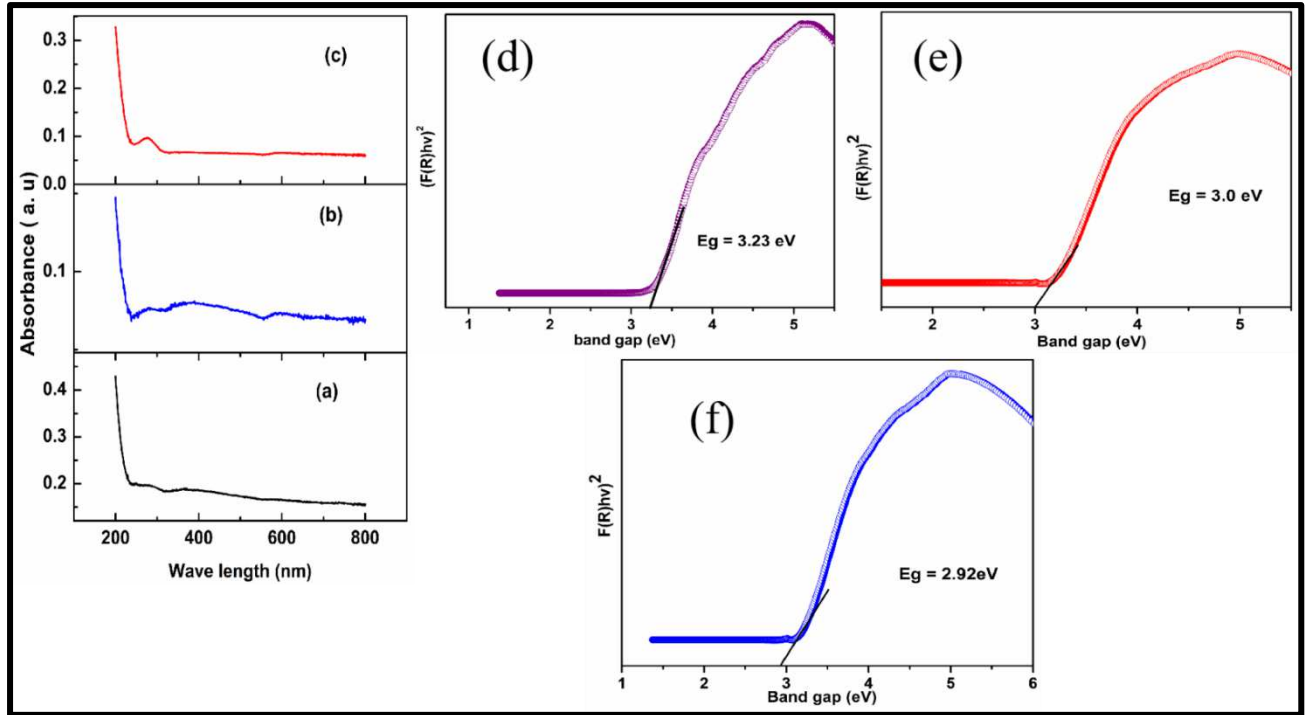


Figure 5. UV-absorbance spectra (a-c) and Tauc's plots (d-f) of $\text{Cd}_{0.05}\text{-CeO}_2$, $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$, $\text{Zr}_{0.1}\text{-Cd}_{0.1}\text{-CeO}_2$ nanocomposites respectively.

4.5. PL-Spectroscopy analysis

The processes of charge transfer, electron-hole recombination, and the presence of structural defects in the synthesized samples can be investigated using photoluminescence (PL) excitation spectroscopy [36]. The PL excitation and emission spectra for CeO_2 nanoparticles doped with Cd^{2+} and co-doped with $\text{Cd}^{2+}/\text{Zr}^{2+}$ are presented in fig.6(A0, A1 and A3). These spectra show a broad emission and excitation bands with distinct peaks observed at 362 nm (violet emission, corresponding to 3.4 eV) [37]. A slight reduction in PL emission intensity is observed as the concentration of Cd^{2+} and $\text{Cd}^{2+}/\text{Zr}^{2+}$ dopants increases. These emission features result from the overlapping of transitions related to the 5d-4f levels of Ce^{3+} ions and defect levels caused by oxygen vacancies, transitioning to O 2p states. Similar broad emission patterns in the 400-470 nm range have been reported by Meng et al., who attributed them to transitions from various defect states to the O 2p orbital [38, 39].

Doping CeO_2 with Cd^{2+} and $\text{Cd}^{2+}/\text{Zr}^{2+}$ induces the formation of oxygen vacancies to preserve charge balance, as Ce^{4+} ions are replaced by lower-valence dopants. This substitution reduces the amount of Ce^{3+} ions required. As dopant levels rise, more oxygen vacancies are generated, while Ce^{3+} content decreases. Consequently, the overall PL emission intensity slightly diminishes with increasing dopant concentration.

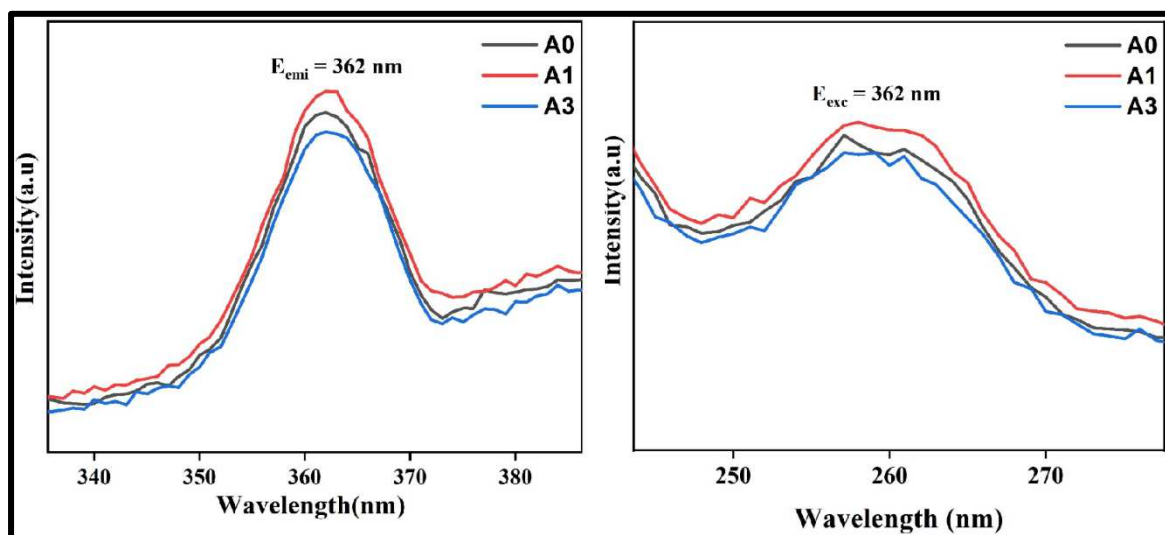


Figure. 6. PL-Excitation and emission spectra of $\text{Cd}_{0.05}\text{-CeO}_2$ (A0) $\text{Cd}_{0.05}\text{-Zr}_{0.05}\text{-CeO}_2$ (A1) $\text{Zr}_{0.1}\text{-Cd}_{0.1}\text{-CeO}_2$ (A3) NPs.

5. Photodegradation

5.1. Photocatalytic activity and dye selection

Upon photon absorption, metal-doped CeO_2 nanoparticles facilitate the excitation of electrons from the valence band to the conduction band, thereby generating electron-hole pairs [40]. The holes migrate from the bulk to the nanoparticle surface, where they oxidize adsorbed H_2O or OH^- species, leading to the evolution of highly reactive hydroxyl radicals ($\bullet\text{OH}$). Concurrently, the conduction band electrons reduce adsorbed O_2 molecules, producing superoxide radicals ($\text{O}_2\bullet^-$). These reactive oxygen species (ROS) subsequently engage in further reactions, including interactions with H_2 , ultimately resulting in the formation of OH and OH^- species [41]. The photocatalytic performance of $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ nanoparticles (10mg) was first evaluated under UV light irradiation using

eight different organic dyes: Rose Bengal, congo red, methylene blue, Evans blue, methyl orange, rhodamine B, indigo carmine, and alizarin as shown in fig.7a. The degradation percentages of these dyes were determined using Equation 1.

Among the seven tested dyes, $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ nanoparticles (10mg) exhibited the highest photocatalytic activity with Rose Bengal, achieving a degradation rate of 72.7%. In contrast, the degradation rates for the other dyes were significantly lower: methylene blue (32.8%), methyl orange (34.4%), indigo carmine (15%), Evans blue (24%), alizarin (45.9%), rhodamine B (64%), and congo red (70%). Due to the superior degradation efficiency observed with Rose Bengal, it was selected for further investigation. Typically, UV-Visible spectroscopy was used to track the reduction in the dye's characteristic absorption peak, which served as an indicator of the degradation process. This trend is depicted in the graph shown in fig 7a.

The photocatalytic efficiency of Cd-CeO_2 , $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$, and $\text{Zr}_{0.1}\text{-Cd}_{0.1}\text{-CeO}_2$ nanoparticles was assessed by evaluating their ability to degrade Rose Bengal (RB) solution during the irradiation UV-light. The observed degradation rates were 69%, 90% and 79.5%, respectively, as illustrated in fig.7 c and fig.7d represents the absorbance of light decreases with time shows the concentration of dye decreases with time through degradation.

5.2. Study about Kinetics

The displayed fig. 7b shows the kinetic analysis of the photodegradation of the dyes using $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ NPs as photocatalyst [40]. The reaction process was a pseudo first order reaction and the rate constant was determined by the equation (4) [42].

$$\ln \frac{C_0}{C_t} = -kt \quad \text{-----} \quad (4)$$

where, C_0 is the initial concentration of the dye solution after illumination, C_t is the concentration of the dye at each 30 minute interval of time after illumination.

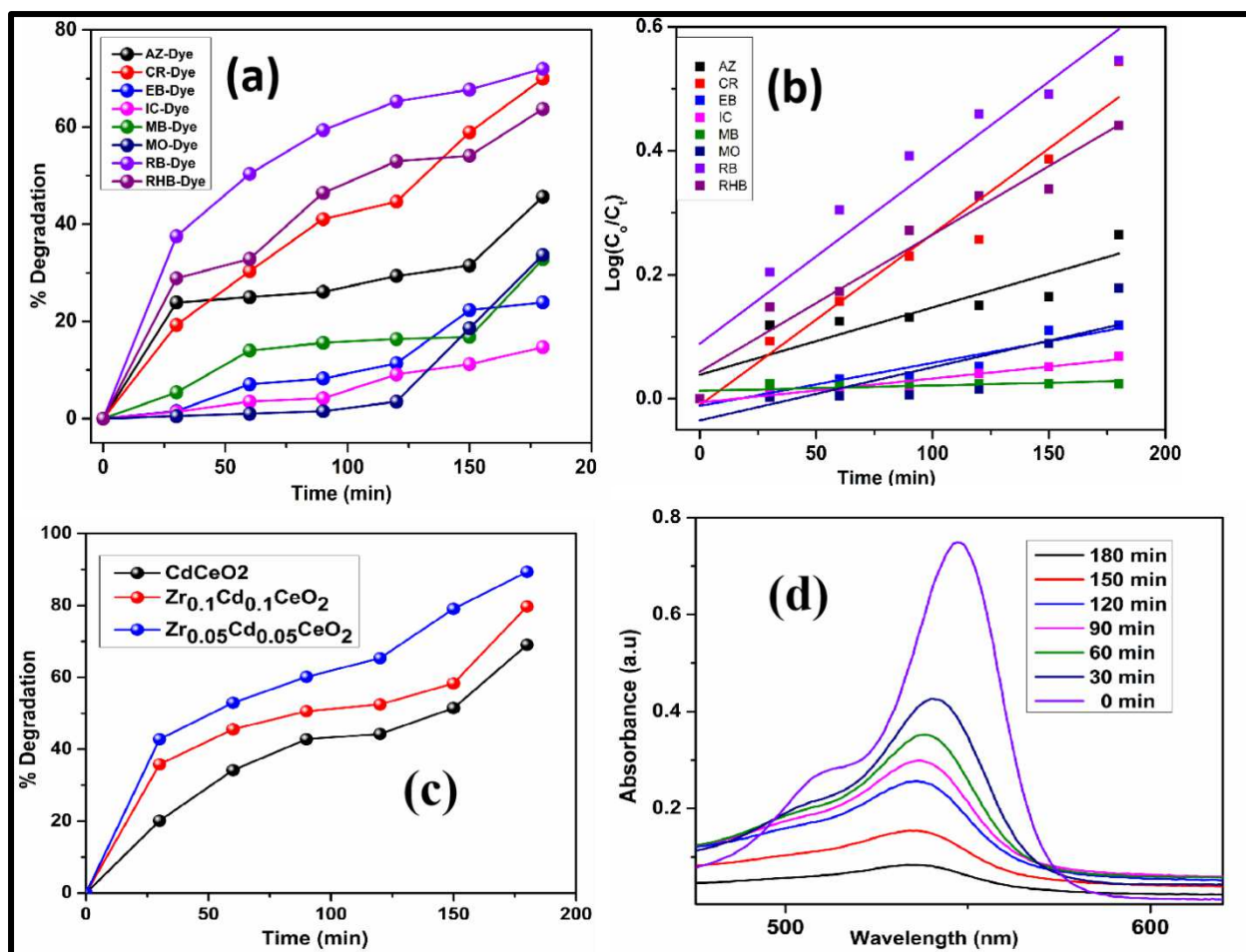


Figure. 7 (a) Selection of dyes (b) Kinetic plots of dye selection (c) % degradation of RB dye by different NPs (d) Degradation of RB dye with time by Zr_{0.05}-Cd_{0.05}-CeO₂ NPs.

The degradation kinetics of each dye were illustrated through the graphical representation provided in fig.7(b), where the slope of each curve corresponds to the respective rate constant (k) of dye degradation. Based on the graph, the calculated rate constants were as follows: 0.00109 min⁻¹ for alizarin, 0.00276 min⁻¹ for carmine red, 0.0000693 min⁻¹ for evans blue, 0.000038 min⁻¹ for indigo carmine, 0.000086 min⁻¹ for methylene blue, 0.0000857 min⁻¹ for methyl orange, 0.00282 min⁻¹ for rose bengal, 0.00221 min⁻¹ for rhodamine B. Among these, the highest k value was observed for rose bengal (0.00282 min⁻¹), indicating that the Zr_{0.05}-Cd_{0.05}-CeO₂ nano photocatalyst exhibited the most efficient photocatalytic activity toward the degradation of this dye. This suggests

that the synthesized nanocomposite serves as an effective photocatalyst, particularly for rose bengal degradation under the applied conditions.

5.3. Effect of photocatalyst dosage

Fig. 8a illustrates that varying the amount of photocatalyst used, from 10 to 40 mg, while maintaining a constant RB concentration of 5 ppm, allows for the evaluation of how different nanocomposite dosages affect the efficiency of RB removal under UV light [43]. Catalyst dosages of 10 mg, 20 mg, 30 mg, and 40 mg were evaluated for their degradation performance. Among these, the 20 mg dosage achieved the highest degradation efficiency at 89.4%. However, as the nanoparticle concentration exceeded 20 mg, a noticeable reduction in degradation efficiency was observed, as shown in the fig. 8a. The outcomes are ascribed to the number of active sites available on the catalyst surface and the extent to which UV-light can penetrate the reaction mixture [44]. The decrease in degradation efficiency at higher catalyst concentrations is primarily due to increased light scattering and shielding effects. When too many photocatalyst particles are present in the suspension, they can obstruct or redirect incoming light, limiting its penetration through the solution. As a result, fewer photons reach the deeper layers of the suspension, leading to a reduced production of electron-hole pairs essential for photocatalytic activity. Additionally, high catalyst loading may lead to particle agglomeration, where particles cluster together rather than remaining uniformly dispersed. This clumping decreases the effective surface area available for reaction, as fewer active sites remain exposed to interact with dye molecules, ultimately hindering the degradation process.

5.4. Effect of dye concentration

The influence of initial concentration of RB dye on nanoparticles performance was investigated. With a fixed amount such as 10 mg of $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ nanoparticles, varying dye concentrations of 5 ppm, 10 ppm, 15 ppm, and 20 ppm were examined to evaluate their effect on the

enhanced photodegradation of dye. The fig.8b demonstrates a steady decrease in photodegradation efficiency, dropping from 89.30% to 59.3% as the dye concentration increases. The higher initial pollutant concentration led to a decrease in the photocatalyst's degradation efficiency, likely due to insufficient interaction between the dye molecules and the active sites on the $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ nanoparticles. This may be caused by either saturation or partial deactivation of these active sites. Additionally, the dense concentration of pollutants can hinder photon penetration, limiting the production of reactive radicals necessary for effective photocatalysis [45]. The findings indicated that increasing dye concentration significantly hindered the productivity of the photodegradation process. This decline can be ascribed to several interconnected factors. One of the main issues is the attenuation of light; as the dye concentration rises, the solution becomes more heavily pigmented, absorbing more of the incident light. This results in reduced light transmission through the solution, limiting its ability to effectively reach the photocatalyst's surface. Since photocatalytic reactions depend on the catalyst being activated by light, reduced light availability means fewer electron-hole pairs are generated, which are essential for forming reactive species such as hydroxyl radicals and superoxide anions. [46]

5.5. Effect of pH

The pH of an organic dye solution is a crucial factor in photolysis, as it influences the charge characteristics of both the surrounding medium and the photocatalysts involved [47]. To determine the optimal pH for photocatalytic activity, a range of pH values from 3 to 11 was tested, maintaining constant conditions with 10 mg of $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ nanoparticles and a 5 ppm concentration of Rose Bengal dye (fig. 8c). The highest catalytic performance of the $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ NPs was observed at pH 3. This acidic condition created an environment conducive to the generation of reactive oxygen species (ROS), thereby enhancing the overall dye degradation efficiency. In acidic media, protonation rendered the surface of the $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ NPs positively charged. This positive

surface charge facilitated stronger electrostatic attraction between the photocatalyst and the negatively charged Rose Bengal dye molecules. As a result, dye adsorption onto the catalyst surface increased, which is a critical preliminary step in photocatalytic degradation. The improved contact under these conditions significantly boosted the degradation process.

Moreover, acidic environments supported the formation and stabilization of ROS such as hydroxyl radicals ($\text{OH}\cdot$), although the yield of these species could be slightly less than in basic conditions. In contrast, under basic conditions, the surface of the photocatalyst became negatively charged, leading to electrostatic repulsion with the anionic dye molecules. This repulsion reduced dye adsorption onto the catalyst, thereby diminishing the degradation efficiency. Although alkaline conditions promoted the formation of hydroxyl radicals due to the abundance of hydroxide ions, the beneficial effect was offset by poor dye-catalyst interaction.

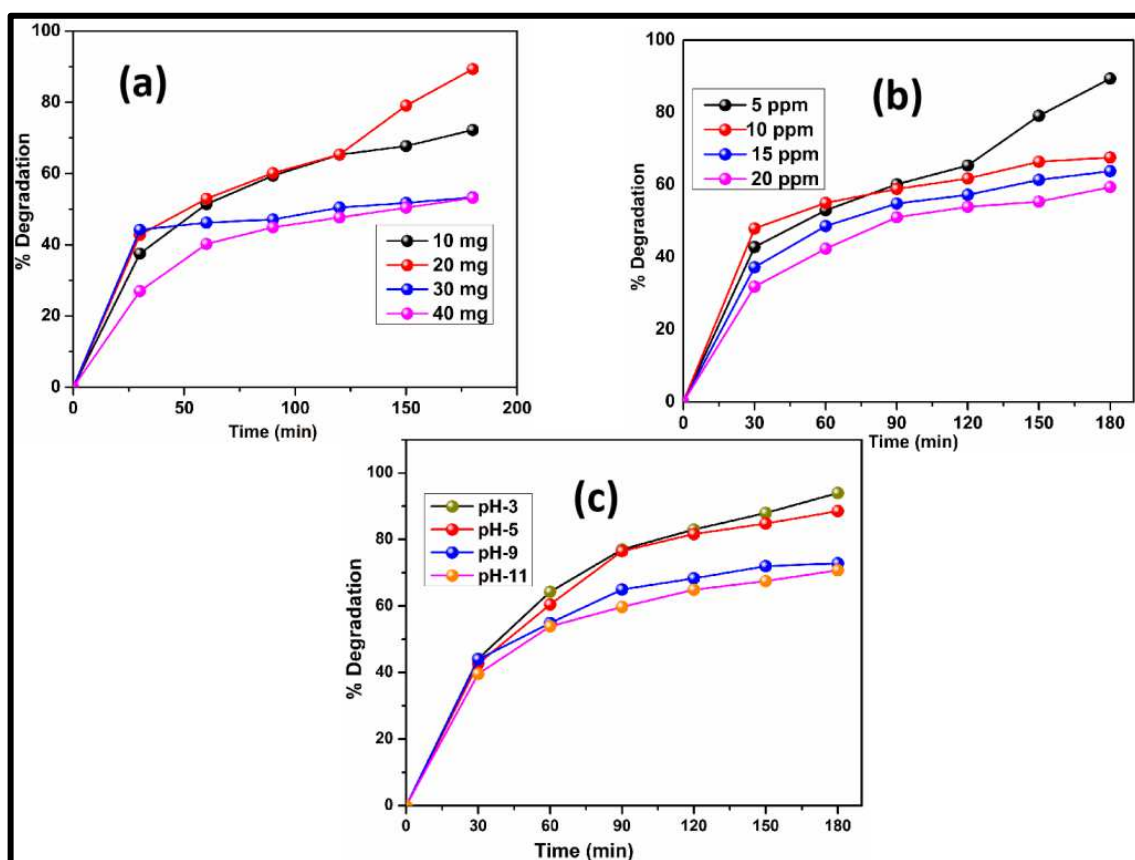


Figure. 8 (a) Degradation of Rose Bengal with varying catalyst load, (b) Degradation of Rose Bengal with varying the concentration of Dye, (c) Degradation of Rose Bengal with varying pH of the solution and keeping the catalyst load and concentration of dye constant.

5.6. Scavengers study and mechanism of degradation

Scavenger experiments were carried out to determine the dominant reactive species involved in the photocatalytic degradation of rose bengal (RB) dye using the $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ photocatalyst. Specific chemical agents were employed to selectively quench different reactive species: potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) for electrons (e^-), tertiary butyl alcohol (TBA) for hydroxyl radicals ($\text{OH}\cdot$), ethylene diamine tetra acetic acid (EDTA) for photogenerated holes (h^+), and ascorbic acid (AA) for superoxide radicals ($\text{O}_2^{\cdot-}$) [48]. The impact of these scavengers on degradation efficiency is illustrated in fig.10a. The inhibition trend observed was as follows: $\text{K}_2\text{Cr}_2\text{O}_7 > \text{AA} > \text{EDTA} > \text{TBA}$. The significant reduction in photocatalytic activity upon addition of $\text{K}_2\text{Cr}_2\text{O}_7$ indicates that electrons are the principal reactive species driving the degradation process. Since these electrons can interact with molecular oxygen to produce superoxide radicals ($\text{O}_2^{\cdot-}$), it confirms their central role in the photodegradation of RB dye when $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ is used.

Although holes (h^+) and hydroxyl radicals ($\text{OH}\cdot$) also contribute to the degradation, their role appears to be secondary. The presence of ascorbic acid, which specifically scavenges superoxide radicals, led to a noticeable decrease in degradation efficiency, further emphasizing the significance of $\text{O}_2^{\cdot-}$ in the overall reaction mechanism. Upon exposure to UV irradiation, the $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ photocatalyst generates electron-hole (e^-/h^+) pairs. The photogenerated electrons in the conduction band reduce adsorbed oxygen molecules to form superoxide radicals, which can either directly oxidize RB dye or react with holes to generate hydroxyl radicals both contributing to the degradation pathway. The mechanism and

graphical representation (fig.9) of degradation of RB dye by photocatalyst as shown below.

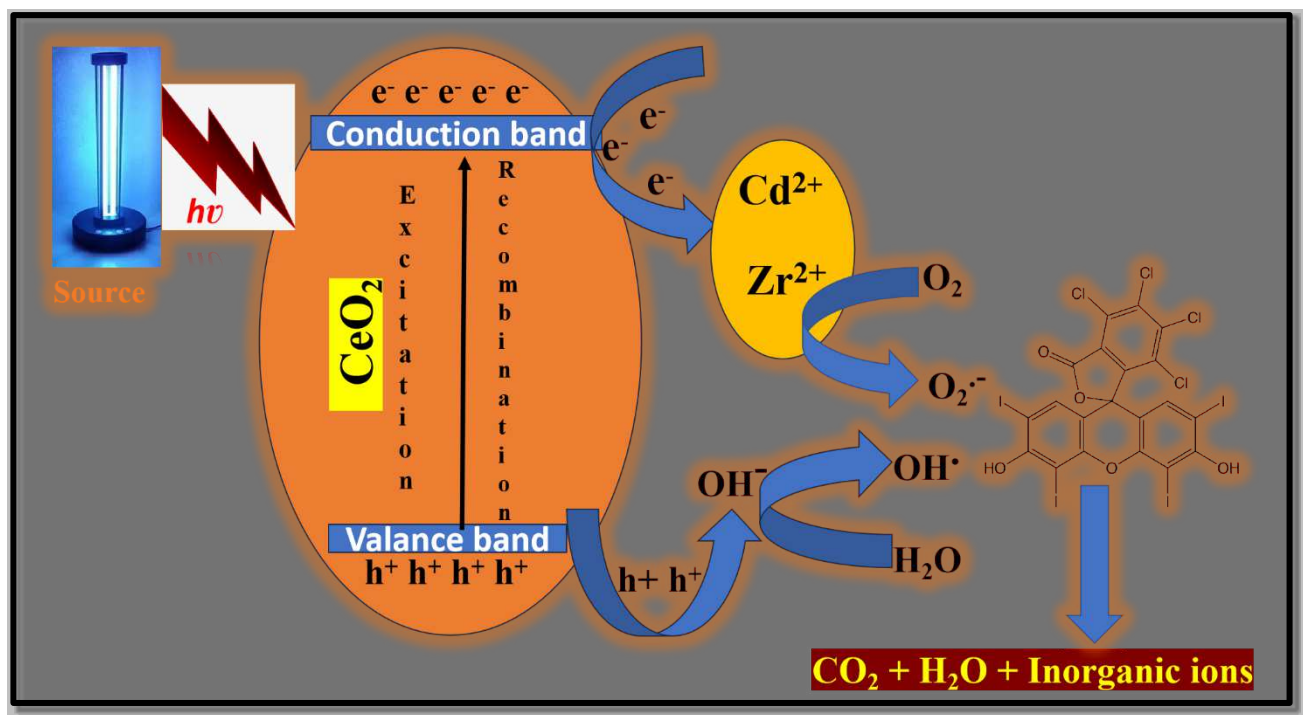
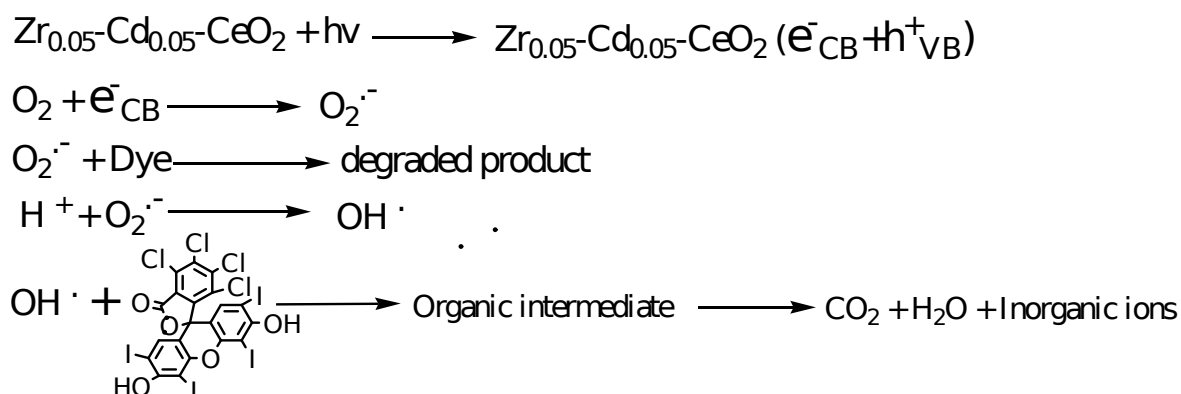


Figure. 9. Graphical representation of photodegradation of RB dye by $\text{Cd}_{0.05}\text{-Zr}_{0.05}\text{-CeO}_2$ NPs.

5.7. Recovery and recycling photocatalyst

In photocatalytic applications, the ability to recover and reuse the catalyst is an important factor for assure both the economic feasibility and environmental sustainability of the degradation process over extended use. To evaluate the reusability of the $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ nanoparticles (NPs), the catalyst was retrieved from the reaction mixture via

centrifugation. The obtained precipitate was thoroughly cleaned with distilled water and ethanol to get rid of any remaining residual dye molecules and surface contaminants. Following the washing procedure, the material was dried in an oven at 80 °C for 3 hours. The regenerated photocatalyst was then reused in subsequent degradation experiments. This recovery and reuse process was repeated across multiple cycles. As illustrated in fig.10b, the photocatalytic efficiency showed minimal decline less than 6% reduction even after four sequential cycles, indicating strong catalytic stability and performance retention.

To further assess the structural unity of the recovered catalyst, X-ray diffraction (XRD) analysis was conducted. The results, shown in the corresponding fig.10c, confirmed that the crystal structure and band gap of the $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ NPs remained intact, demonstrating excellent crystallinity and structural robustness after repeated use.

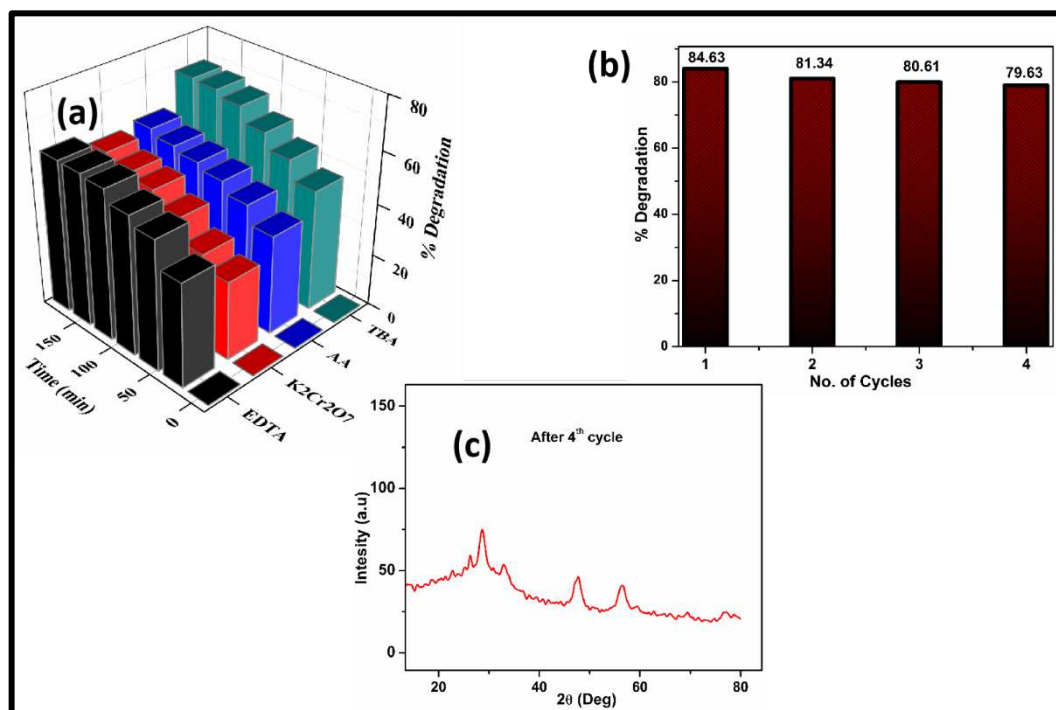


Figure.10. (a) Scavengers studies in Degradation process of Rose Bengal using $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ NPs. (b) Degradation efficiency of 10mg $\text{Cd}_{0.05}\text{-Zr}_{0.05}\text{-CeO}_2$ NPs (c) p-XRD spectra of recycled $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ NPs.

5.8. Reusing of photodegraded water in plant growth

Fenugreek, known for its sensitivity and rapid growth, was selected to assess the impact of dye-contaminated water and its treated forms on plant development, both before and after the photodegradation of Rose Bengal (RB) dye using nanoparticles [49]. Four days after germination, growth was monitored in seeds cultivated in various water samples, including untreated dye water, photo-degraded wastewater, and distilled water. The results illustrated in fig.11 demonstrate that plants irrigated with photo-degraded wastewater showed growth patterns nearly identical to those grown in distilled water. This suggests that the degradation process significantly reduced dye toxicity, thereby minimizing its interference with photosynthesis and allowing normal plant development. In contrast, fenugreek growth was significantly inhibited in untreated dye-contaminated water. As depicted in fig.12 (a-b), vigorous growth of roots and shoots was observed in distilled water, while photo-treated water also supported healthy development, indicating that the toxic components had been effectively neutralized. Thus, the use of nanomaterials for the photodegradation of dye-contaminated water resulted in detoxified effluents that supported effective plant growth, comparable to that seen with clean, distilled water.



Figure.11. Growth of fenugreek plant in different waters

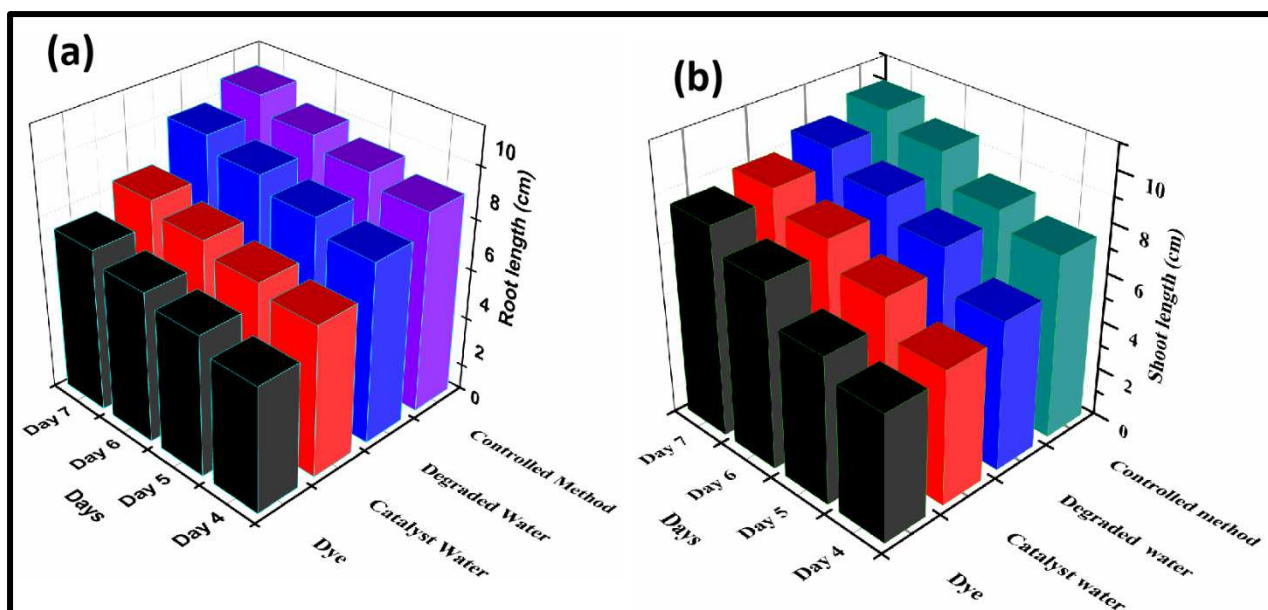


Figure.12. Root length (a) and shoot length (b) of fenugreek plants.

6. Latent Finger print applications of $\text{Cd}_{0.05}\text{-Zr}_{0.05}\text{-CeO}_2$ NPs

Human fingerprints serve as one of the desperately distinctive and reliable forms of identification, playing a crucial role in forensic enquiry. Latent fingerprints (LFPs) are among the most normally used methods in criminal casework. Multiple metal deposition, powder dusting, Raman spectroscopy, ninhydrin dipping, plasmonic imaging, iodine fuming, fluorescence spectroscopy, silver nitrate soaking, and more have all been used to visualize LFPs. The powder dusting technique is thought to be the best among them for improving LFP visibility because it makes the essential ridge details visible [50, 51, 52]. As seen in fig.13a,b, latent fingerprints (LFPs) were formed on hard surfaces such a glass plate and a cell phone screen. The collected fingerprints displayed a multiple characteristic patterns, including eyes, loops, whorls, arches, fork, bifurcation, ulnar loop, short ridge etc (fig.14 a-g) are the common features used in biometric identification. Notably, the ridge details within the fingerprints were clearly visible and well-defined, without any distortion or background interference. This high level of clarity is crucial for accurate forensic analysis and identification. The successful visualization of these ridge patterns highlights the effectiveness and

potential applicability of the synthesized nanoparticles (NPs) in LFP detection and forensic investigations.

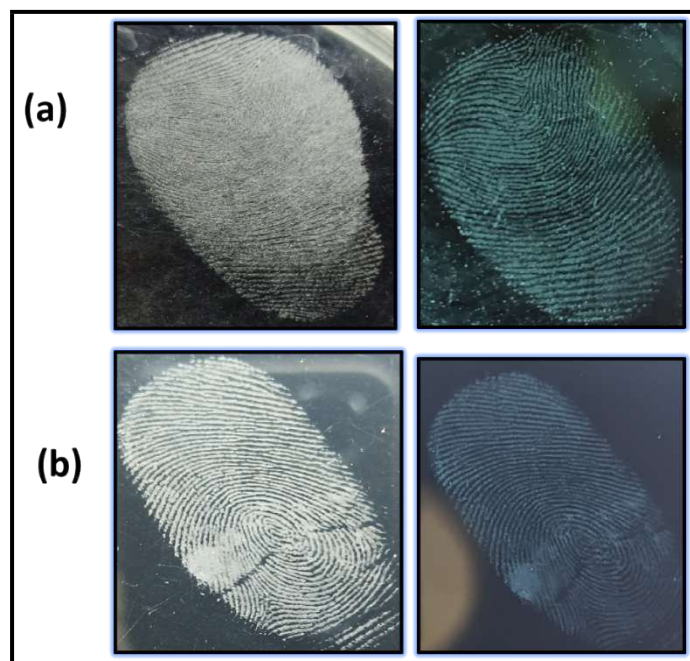


Figure.13. Impression on smooth surfaces developed using $Zr_{0.05}-Cd_{0.05}-CeO_2$ NPs on (a) petri plates and Mobile screen (b).

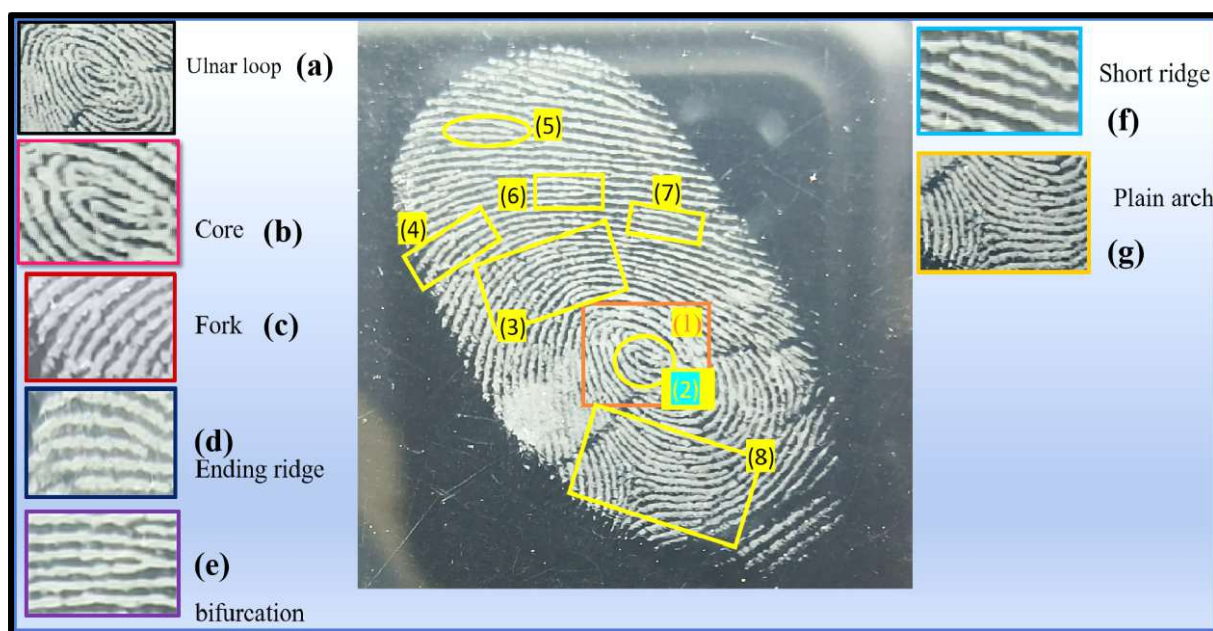


Figure. 14. Identifying (a) Ulnar loop (b) core (c) fork (d) ending ridge (e) bifurcation (f) short ridge (g) plain ridge in finger print developed by $Zr_{0.05}-Cd_{0.05}-CeO_2$ NPs.

7. Conclusion

In this study, Cd-doped and Zr/Cd co-doped CeO₂ nanoparticles were synthesized via a green solution combustion method utilizing *Lobularia maritima* seed extract. The as-prepared nanomaterials were systematically characterized using a range of analytical techniques, including powder X-ray diffraction (p-XRD), Fourier-transform infrared spectroscopy (FT-IR), scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDX), UV-visible diffuse reflectance spectroscopy (UV-DRS), and photoluminescence (PL) spectroscopy. The optical band gap energies for Cd-doped CeO₂, 5 mol% Zr and Cd co-doped CeO₂, and 10 mol% Zr and Cd co-doped CeO₂ nanoparticles were found to be 3.23 eV, 3.00 eV, and 2.92 eV, respectively. FT-IR spectra confirmed the presence of key functional groups, while XRD patterns revealed high phase purity and crystallinity of the synthesized materials. The average crystallite sizes, calculated using the Scherrer equation, were determined to be 23.4 nm for Cd_{0.05}-CeO₂, 21 nm for Zr_{0.05}-Cd_{0.05}-CeO₂, and 18 nm for Zr_{0.1}-Cd_{0.1}-CeO₂ nanoparticles. SEM analysis indicated a predominantly spherical morphology with mesoporous characteristics. EDX confirmed the elemental composition of the nanomaterials, detecting Ce, Cd, Zr, and O as primary constituents, along with minor traces of N and C. PL spectroscopy suggested that the incorporation of Cd²⁺ and Zr²⁺ ions into the CeO₂ lattice induced oxygen vacancies, which play a critical role in enhancing photocatalytic activity. Among the synthesized catalysts, Zr_{0.05}-Cd_{0.05}-CeO₂ exhibited the highest photocatalytic efficiency, achieving approximately 90% degradation of the organic dye Rose Bengal (RB), compared to 60% for Cd-CeO₂ and 79.5% for Zr_{0.05}-Cd_{0.05}-CeO₂ under identical conditions. The optimal parameters for efficient dye degradation using Cd_{0.05}-Zr_{0.05}-CeO₂ were identified as 20 mg catalyst dosage, 5 ppm dye concentration, and pH 3. The catalyst demonstrated excellent stability and reusability, retaining significant photocatalytic activity after four consecutive degradation cycles. Reactive species trapping experiments confirmed that hydroxyl radicals were the predominant active species responsible for the photodegradation of RB. Furthermore, treated dye wastewater was reused in a plant growth experiment involving *Trigonella*

foenum-graecum (fenugreek), which showed healthy growth comparable to that irrigated with clean water, indicating the non-toxicity of the treated effluent. Finally, due to its nanoscale crystallite size and favorable physicochemical properties, the $\text{Zr}_{0.05}\text{-Cd}_{0.05}\text{-CeO}_2$ nanocomposite was successfully employed in the visualization of latent fingerprints, demonstrating its potential application in forensic science.

8. Statement of authorship contribution:

Suresha E: Conceptualisation, Writing original draft, Software and Methodology, S Sreenivasa : Supervision and Investigation, Methodology, . G. Nagaraju: Conceptualization, Investigation and resources. Pooja : Data curation, methodology and review.

9. Declaration of Competing Interest:

The authors declare that they have non known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

10. Data availability:

Data will be made available on report.

11. Funding information

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