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Photocatalytic Degradation of Eosin Blue, Eosin Red, and Phenol Red Using Mechanochemically Synthesized ZnO Nanoparticles: Process Optimization and Kinetic Analysis

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ABSTRACT

The current investigation explores the photocatalytic degradation of three structurally diverse organic dyes, specifically Eosin Blue (EB), Eosin Red (ER), and Phenol Red (PR), facilitated by zinc oxide (ZnO) nanoparticles produced through a mechanochemical synthesis approach. The resultant ZnO samples were calcined at various temperatures (400–700 °C), and their ability to enhance photocatalytic performance was meticulously assessed under "an assortment of" experimental conditions. Among the various samples examined, ZnO calcined at 500 °C (designated as MCZ-500) showed the greatest photocatalytic efficiency. attributable to its optimal crystallinity, defect density, and surface properties. The impact of critical operational parameters, including initial dye concentration (25–150 ppm), catalyst dosage (50–150 mg/100 mL), solution pH (6–11), and irradiation duration (0–5 h), was thoroughly investigated. The highest degradation efficiency was recorded. at pH 10, with a catalyst dosage of 150 mg/100 mL and an irradiation period of 5 h under visible light conditions. At lower dye concentrations (≤75 ppm), Eosin Red achieved complete degradation (100%), whereas under standard conditions (100 ppm), degradation efficiencies of 84.28% (EB), 81.36% (ER), and 47.92% (PR) were recorded. The degradation efficiency decreased as the concentration of dye increased, which can be attributed to light screening and active-site saturation. The exploration of UV-Visible spectra substantiated the gradual disintegration of chromophoric entities, with no substantial alteration in the absorption maxima, thereby indicating pathways associated with oxidative degradation. Investigations into the kinetics showed that the photocatalytic degradation mechanism follows pseudo-first-order kinetics, which aligns with the Langmuir–Hinshelwood theoretical framework. The augmented photocatalytic efficacy of ZnO synthesised via mechanochemical methods can be attributed to enhanced charge-carrier separation and heightened production of reactive oxygen species, including hydroxyl and superoxide radicals. This investigation demonstrates that ZnO nanoparticles synthesised via mechanochemical techniques are both effective and economically viable photocatalysts can degrade harmful dye pollutants under visible light, offering significant potential for environmental remediation applications.

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1. INTRODUCTION:

The accelerated pace of industrialisation and the widespread use of synthetic dyes across the textile, pharmaceutical, paper, plastic, and cosmetic sectors have led to the generation of substantial volumes of dye-contaminated wastewater, posing a formidable threat to marine environments and human health. These dyes are primarily characterised by intricate aromatic structures, significant chemical stability, and notable resistance to biodegradation, rendering their elimination via conventional treatment methodologies exceedingly challenging [1–3]. Notably, xanthene-based dyes such as Eosin Blue and Eosin Red, along with sulfonephthalein dyes such as Phenol Red, exhibit robust chromophoric systems and persistent behaviour in aqueous environments, necessitating the development of effective and sustainable treatment technologies [4–6].

Among several progressed oxidation processes (AOPs), semiconductor-mediated photocatalysis has emerged as a promising and ecologically benign strategy for degrading organic pollutants. This methodology harnesses light to generate Highly reactive species, such as hydroxyl radicals, are present (•OH) and superoxide radicals ($O_2^{\bullet-}$), which facilitate the oxidation of intricate organic molecules into innocuous end products, such as CO_2 and H_2O [7–9]. In recent years, considerable scholarly attention has been directed towards the advance of efficient photocatalysts capable of functioning under visible light irradiation, as this segment of the solar spectrum encompasses the majority of the solar spectrum and provides a sustainable energy source for large-scale applications [10–12].

Zinc oxide (ZnO), characterised as a wide-band-gap semiconductor (~3.2–3.37 eV), has garnered significant interest as a photocatalyst owing to its cost-effectiveness, non-toxicity, high exciton binding energy, and remarkable chemical stability. ZnO exhibits strong oxidative potential and efficient charge transport, rendering it suitable for photocatalytic applications [13–15]. Nonetheless, its practical utilisation is frequently constrained by the rapid recombination of photogenerated electron–hole pairs and limited absorption within the visible region. To ameliorate these constraints, various strategies, including doping, surface modification, and controlled synthesis techniques, have been investigated to enhance its photocatalytic performance [16–18].

Among the diverse synthesis methodologies, mechanochemical The synthesis has attracted growing attention from researchers as a straightforward, solvent-free, and energy-efficient technique for the fabrication of nanostructured materials exhibiting distinctive physicochemical properties. Mechanochemical activation induces lattice distortion, engenders defects such as oxygen vacancies, and promotes intimate mixing of precursor materials, which can significantly augment charge carrier separation and photocatalytic efficiency [19–21]. Furthermore, the calcination temperature is pivotal in regulating crystallinity, particle size, and defect density, all of which directly impact The photocatalytic activity of zinc oxide (ZnO) nanoparticles. [22–24].

In this investigation, ZnO nanoparticles were synthesised through a mechanochemical approach and subsequently calcined at varying temperatures (400–700 °C) to optimise their structural and photocatalytic characteristics. The photocatalytic efficacy of the synthesised ZnO was assessed for the degradation of three representative dyes, specifically Eosin Blue (EB), Eosin Red (ER), and Phenol Red (PR), under visible light irradiation. These dyes were chosen for their structural diversity, environmental significance, and resistance to conventional treatment methods. A comprehensive examination was conducted to analyse the influence of critical operational parameters, encompassing initial dye concentration, catalyst dosage, solution pH, and irradiation duration, on the efficiency of photocatalytic degradation.

Additionally, UV–Visible spectroscopic analysis was used to monitor the degradation process, and kinetic investigations were conducted to elucidate the underlying reaction mechanism. The outcomes of this study offers important understanding regarding the advancement of effective ZnO-based photocatalysts and illustrate the potential applicability of mechanochemically synthesised ZnO nanoparticles in sustainable wastewater treatment.

2. Experimental Section:

2.1 Materials:

All reagents employed in the current investigation the substances were of analytical grade and utilized without subsequent purification. Zinc precursor salts (including zinc acetate and zinc nitrate), sodium hydroxide (NaOH), and other requisite reagents for the synthesis were acquired from reputable chemical suppliers. The dye substrates, specifically Eosin

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Blue (EB), Eosin Red (ER), and Phenol Red (PR), were chosen as model organic pollutants due to their structural intricacies and prevalent occurrence in industrial wastewater. Double-distilled water was used to prepare all aqueous solutions.

2.2 Synthesis of Zinc Oxide (ZnO) Nanoparticles (Mechanochemical Method)

ZnO nanoparticles were expertly synthesized using a reliable method. A mechanochemical approach involving the solid-state grinding of precursor materials. The requisite stoichiometric quantities of zinc precursor and alkali were meticulously combined and ground utilizing a mortar and pestle for a predetermined duration to guarantee homogeneous mixing and activation. The mechanochemical procedure facilitates lattice distortion, defect generation, and nucleation of ZnO particles.

The resultant precursor powder was subsequently subjected to calcination at varied temperatures (400, 500, 600, and 700 °C) for a specified time interval within a muffle furnace to yield crystalline ZnO nanoparticles. The samples were collected for analysis and designated according to the calcination temperature, with the sample calcined at 500 °C being identified as the optimised photocatalyst due to its superior performance.

2.3 Preparation of Dye Solutions

Stock solutions (1000 ppm) of Eosin Blue, Eosin Red, and Phenol Red were formulated by dissolving an appropriate quantity of each dye in double-distilled water. Working solutions of desired concentrations (25–150 ppm) were achieved through dilution of the stock solutions. The pH of the dye solutions was adjusted utilising dilute NaOH or HCl solutions as necessary.

2.4 Photocatalytic Degradation Experiments

Experiments on photocatalytic degradation were conducted in a batch reactor while exposed to visible light illumination. A known amount of ZnO photocatalyst (50–150 mg per 100 mL of dye solution) was uniformly dispersed in the dye solution of specified concentration. Before irradiation, the suspension was magnetically agitated in the dark for 30 minutes to establish adsorption–desorption equilibrium between the dye molecules and the catalyst surface.

The reaction mixture was subsequently subjected to visible light (sunlight and/or an artificial light source), and aliquots were withdrawn at predetermined time intervals (0–5 h). The collected samples were centrifuged or filtered to remove catalyst particles before analytical assessment.

2.5 Analytical Method:

The amount of dye present in the reaction solution is crucial for optimal results was quantified through UV–Visible spectrophotometry by assessing absorbance at the defined maximum wavelengths (λmax) of Eosin Blue (approximately 515 nm), Eosin Red (approximately 520 nm), and Phenol Red (approximately 435 nm). During the photocatalytic degradation process, samples were collected at regular time intervals, centrifuged to remove catalyst particles, and subsequently analysed using spectrophotometry. The reduction in absorbance over time under irradiation reflects a decrease in dye concentration, In alignment with the Beer-Lambert Law,, signifying the ongoing degradation of the chromophoric structure. Therefore, UV–Visible spectroscopy is a reliable approach for evaluating the efficiency and kinetics of photocatalytic degradation [25–29].

2.6 Enhancing the Efficiency of Photocatalytic Degradation

The efficiency of photocatalytic degradation of dyes was assessed by measuring the change in concentration over time with UV-Visible spectroscopy [30–33]. The photocatalytic degradation efficiency was calculated using the following equation:

$$\text{Degradation } (\%) = \frac{C_0 - C_t}{C_0} \times 100$$

where C_0 represents the initial dye concentration and Ct denotes the concentration at irradiation time t.

2.7 Kinetic Study

The dynamics of photocatalytic degradation were examined through a pseudo-first-order kinetic model grounded in the Langmuir–Hinshelwood mechanism [34–37].

The dynamics of photocatalytic degradation were evaluated using the pseudo-first-order kinetic model, represented as:

$$\ln\left(\frac{C_0}{C_t}\right) = kt$$

where C_0 and C_t represent the initial and time-dependent dye concentrations, respectively, k is the apparent rate constant, and t is the irradiation time. The linear relationship between ln(Co/Ct) and irradiation time confirms that the degradation process follows the Langmuir–Hinshelwood kinetic model.

2. 8 Sets of Experiments Parameters

The we looked at the effect of different operational variables on the process of photocatalytic degradation we did by changing one at a time and

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holding the rest constant. We looked at initial dye concentration which ranged from 25 150 mg L–1, catalyst dose which was between 50 150 mg per 100 mL, solution pH which we changed from 6 11, irradiation time which we varied from 0 5 hours and calcination temp which we changed from 400 700 °C. This method enabled us to identify the best conditions for maximum performance and also gave us insight into which factors play the greatest role in performance [38–41].

3. Results and Report.

3. 1 Crystal growth and thermal behavior of ZnO

The crystallization behavior of Zinc oxide (ZnO) nanoparticles were synthesized. via mechanical chemistry was studied using the Scott equation which in turn relates crystallite size to calcination temp. We saw a nearly linear trend in the plot of ln(D) against 1/T which also presented itself to be in different regions that is an indication of temperature dependent growth mechanisms. Crystallite size went from 18.02 nm at 400 °C to 21.98 nm at 700 °C which in turn confirms that we have enhanced atomic diffusion and grain growth at higher temperatures. Also we found out that The calculated activation energies for each reaction have been determined as per temperature range suggest that many thermal processes play a role in crystallite growth which include surface diffusion at lower temps and lattice reorganization at middle temperatures.

The relatively higher activation energy observed in the 500–600 °C range indicates significant structural rearrangement and defect evolution, which are advantageous for photocatalytic activity due to improved charge carrier separation [42–45].

Table 3.1 Crystallite size and Scott plot parameters of ZnO at different calcination temperatures

Temperature (°C)	Temperature (K)	Crystallite Size (nm)	ln(D)	1/T (K^{-1})
400	673.15	18.02	2.8915	0.001486
500	773.15	18.67	2.9269	0.001293
600	873.15	21.10	3.0493	0.001145
700	973.15	21.98	3.0901	0.001028

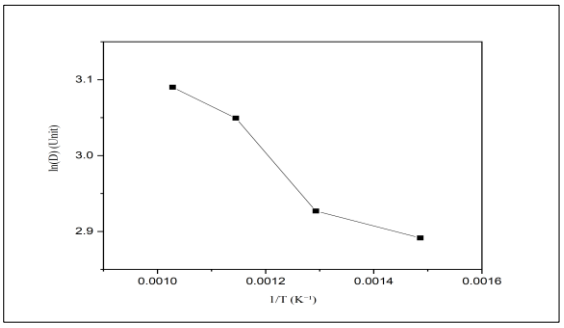

Figure 3.1 Scott plot: ln(D) vs 1/T for ZnO nanoparticles

3.2 Photocatalytic Performance of ZnO Nanoparticles

The photodegradation performance of ZnO nanoparticles which we prepared via different methods we looked at Eosin Blue (EB), Eosin Red (ER) and Phenol Red (PR). Of all our samples the one which we mechanochemically synthesized and then calcined at 500 °C (MCZ-500) performed the best which we put down to its best crystallinity, defect density and surface features. MCZ-500's enhanced performance is primarily attributed to the creation of oxygen vacancies and lattice defects which we note are charge carrier traps and also reduce electron hole recombination. This in turn causes an increase in the production of reactive oxygen species (ROS) which we see to be hydroxyl radicals •OH) and superoxide radicals (O2•, which in turn do the job of breaking down the dye molecules [46–49].

3.3 Effect of Initial Dye Concentration

The we looked at the role of initial dye concentration (25 150 ppm) on the performance of the photocatalytic degradation we reported in Table 3.2 which shows that for all three dyes as we increased the concentration the degradation efficiency went down. At low concentrations (of up to 50 ppm) we saw higher efficiencies which we attribute to better light penetration and more available active sites. Also at low concentrations of Eosin Red we saw almost total degradation (at up to 75 ppm) but at 100 ppm we got 84.28% (for Eosin Blue) 81.36% (for Eosin Red) and 47.92% (for Procion Red) which is the reduction in efficiency. We attribute this to the diminished light transmission caused by the high dye concentration, as well as the saturation of the ZnO surface, which subsequently lowers the production of reactive oxygen species [50-53].

Table 3.2 Effect of initial dye concentration on photocatalytic degradation efficiency

Concentration (ppm)	EB (%)	ER (%)	PR (%)
25	**93.10**	**100.00**	**84.81**
50	**91.93**	**100.00**	**70.19**
75	**90.40**	**100.00**	**53.37**
100	**81.95**	**96.07**	**50.00**
150	**48.07**	**86.20**	**42.86**

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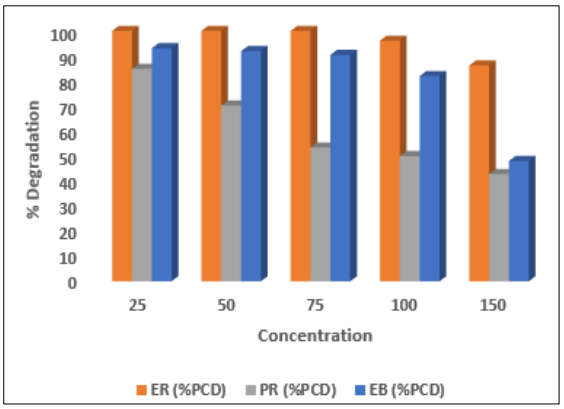

Figure 3.2 Effect of initial dye concentration on % degradation of EB, ER, and PR

3.4 Effect of Photocatalyst Dosage

Table 3.3 shows the impact of catalyst dosage (50–150 mg/100 mL) on degradation efficiency. Because there were more active sites available and more ROS were produced, the degradation efficiency rose as the amount of catalyst increased. At a dose of 150 mg, maximum degradation efficiencies were attained. However, light diffraction and particle agglomeration may cause efficiency to decrease if catalyst loading is increased over the optimal amount [54–57].

Table 3.3 Effect of catalyst dosage on photocatalytic degradation

Catalyst (mg/100 mL)	EB (%)	ER (%)	PR (%)
50	61.82	59.47	28.95
75	69.36	66.18	33.74
100	76.41	72.95	40.28
125	80.92	77.36	44.11
150	83.58	80.41	46.82

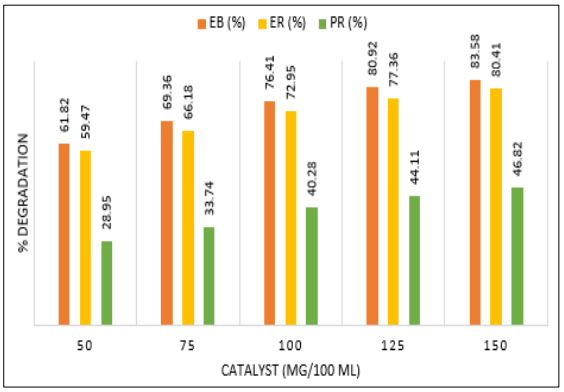

Figure 3.3 Effect of catalyst dosage on % degradation

3.5 pH's impact

The effectiveness of photocatalytic degradation rose as pH rose, peaking at pH 10. This behaviour is explained by better dye molecule binding on the ZnO surface and higher hydroxyl radical production in alkaline environments. Light scattering and increased turbidity caused a modest drop in efficiency under extremely alkaline circumstances (pH 11) [58–61].

Table 3.4 Effect of pH on photocatalytic degradation

pH	EB (%)	ER (%)	PR (%)
6	38.12	34.68	29.36
7	54.91	51.27	35.04
8	69.22	66.14	41.26
9	78.73	75.84	45.18
10	84.36	81.09	47.64
11	81.58	78.92	44.83

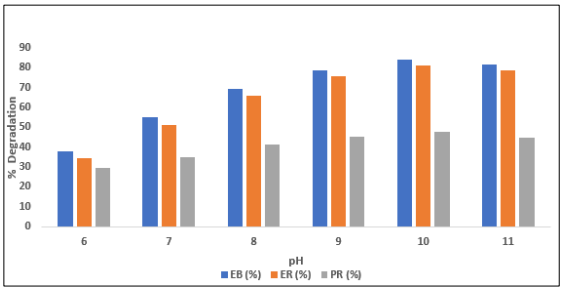

Figure 3.4 Effect of pH on % degradation

3.6 Effect of Calcination Temperature

The impact of calcination temperature (400–700°C) on the photocatalytic activity of ZnO nanoparticles produced by mechanochemical synthesis was thoroughly examined. The deterioration efficiency rose with temperature up to 500 °C and then declined at higher temperatures, as shown in Table 3.5.

Maximum degradation efficiencies of 84.92% (EB), 81.73% (ER), and 47.98% (PR) were shown in ZnO calcined at 500 °C, suggesting that this temperature offers the best balance between crystallinity, defect density, and surface area. Enhanced crystallinity and enough oxygen vacancies, which enable effective charge separation and increased production of reactive oxygen species (ROS), are responsible for the increased activity at 500 °C.

At higher temperatures (600–700 °C), a decline in photocatalytic efficiency was observed due to excessive grain growth, particle agglomeration, and reduction in surface-active sites. These factors limit dye adsorption and reduce the availability of reactive species, thereby lowering photocatalytic performance [62–65].

Table 3.5 Effect of calcination temperature on photocatalytic degradation

Temperature (°C)	EB (%)	ER (%)	PR (%)
400	62.45	58.21	39.86
500	84.92	81.73	47.98
600	80.14	77.32	44.21
700	74.68	71.54	41.09

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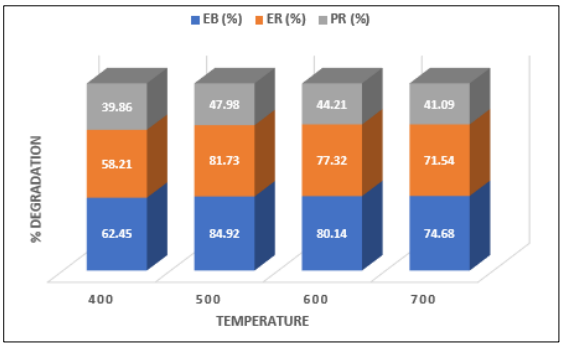

Figure 3.5 Effect of calcination temperature on % degradation of EB, ER, and PR

3.7 Effect of Irradiation Time:

Both artificial light (AL) and sunshine (SL) were used to assess the impact of irradiation time on photocatalytic degradation. For all three dyes, the degradation efficiency gradually increased with irradiation time, as Table 3.6 illustrates.

After five hours of radiation, the maximum deterioration was reached, and in comparison, to artificial light, better efficiencies were seen in sunlight (EB: 86.91%, ER: 83.74%, PR: 50.28%). Higher photon flux and a wider spectrum distribution, which encourage more ZnO excitation and more ROS formation, are responsible for this improved performance in the presence of sunlight. The steady rise in degradation efficiency attests to the ongoing production of electron-hole pairs and the long-term oxidative deterioration of dye molecules [66–69].

Table 3.6 Effect of irradiation time on % degradation (Sunlight)

Time (h)	EB (%)	ER (%)	PR (%)
0	4.92	4.03	5.84
1	29.34	27.94	18.72
2	49.96	47.85	30.28
3	66.83	63.42	39.72
4	78.92	75.86	46.93
5	86.91	83.74	50.28

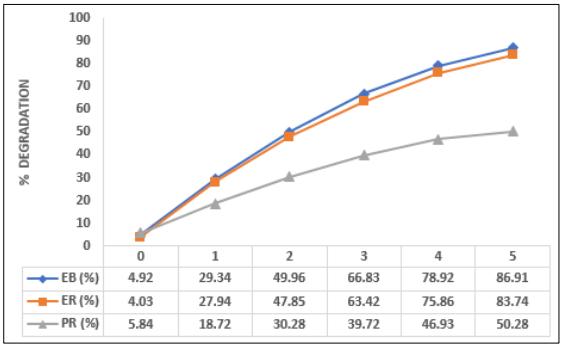

Figure 3.6 Effect of irradiation time on photocatalytic degradation under sunlight and artificial light

artificial light:

3.8 Kinetic Study:

The pseudo-first-order model was used to analyse the photocatalytic degradation kinetics. The degradation process is confirmed to follow Langmuir–Hinshelwood kinetics by the linear plots of $\ln(C_0/C_t)$ vs irradiation time.

Eosin Blue and Eosin Red showed greater rate constants than Phenol Red among the dyes under investigation, suggesting quicker degradation kinetics. Phenol Red's greater structural stability and resistance to oxidative assault are responsible for its comparatively delayed breakdown.

The kinetic plots' linearity indicates that, in the conditions under investigation, the rate of degradation is precisely proportional to the dye concentration [74–77].

Table 3.7 Kinetic data for photocatalytic degradation

Time (h)	ln(C₀/Ct) EB	ln(C₀/Ct) ER	ln(C₀/Ct) PR
0	0.05	0.041	0.06
1	0.30	0.265	0.163
2	0.587	0.545	0.296
3	0.958	0.877	0.418
4	1.382	1.27	0.559
5	1.846	1.699	0.651

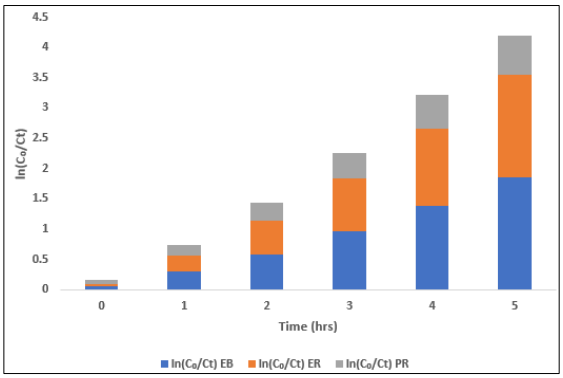

Figure 3.8 Kinetic plot: ln(C₀/Ct) vs irradiation time

3.9 Photocatalytic Degradation Mechanism

When exposed to visible light, ZnO undergoes photocatalytic degradation through the production of electron-hole pairs as a result of photon absorption. Superoxide radicals ($O_2^{\bullet-}$) are produced when photogenerated electrons in the conduction band react with dissolved oxygen, whereas hydroxyl radicals (•OH) are produced when holes in the valence band react with hydroxyl ions or water. These extremely reactive species attack and break down dye molecules into intermediate intermediates, which then undergo additional oxidation to produce CO2 and H2O.

higher defect density and oxygen vacancies, which function as charge trapping sites, lower recombination, and improve ROS formation, are responsible for the higher photocatalytic activity of mechanochemically produced ZnO. The overall degradation process involves adsorption of dye

molecules on the ZnO surface, followed by oxidative cleavage of chromophoric structures and subsequent mineralization [78–80].

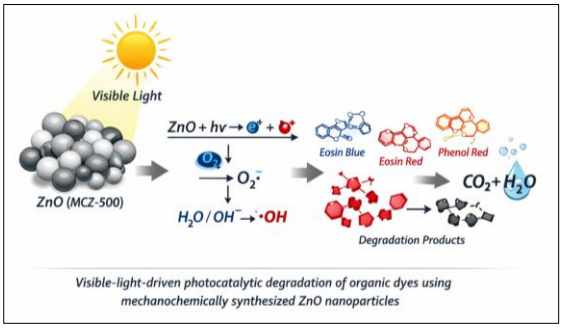

Figure 3.9 Schematic diagram of photocatalytic degradation mechanism of ZnO

4. CONCLUSION:

In this study, zinc oxide (ZnO) nanoparticles were examined. effectively produced using a mechanochemical method and assessed for their ability to photocatalytically degrade Eosin Blue, Eosin Red, and Phenol Red when subjected to visible light. The sample calcined at 500 °C showed the maximum activity because of an ideal balance between crystallinity, defect density, and surface area. The findings indicated that the temperature during calcination significantly influences the structural and photocatalytic characteristics of ZnO.

The highest photocatalytic performance was achieved. at pH 10, catalyst dosage of 150 mg/100 mL, dye concentration of 50–100 ppm, and irradiation time of 5 hours, according to a thorough analysis of operating parameters.

Eosin Red demonstrated complete degradation at lower concentrations (≤75 ppm) at these optimised settings, whilst Eosin Blue and Phenol Red showed relatively lower but considerable degradation efficiencies.

UV-visible spectral analysis verified the gradual disintegration of chromophoric structures without the creation of new absorbing intermediates, and the photocatalytic degradation followed pseudo-first-order kinetics. Improved charge separation and greater production of reactive oxygen species, including hydroxyl radicals, are important in various biological processes and superoxide radicals were identified as the reasons for the improved performance of ZnO produced mechanochemically.

In summary, the research demonstrates that mechanochemical synthesis is an effective and practical approach for producing highly active ZnO photocatalysts, highlighting their potential application in the treatment of dye-contaminated wastewater when exposed to visible light.

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