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Impact of Laser Scanning Area on Zinc Oxide Nanoparticle Formation via Pulsed Laser Ablation in Liquid and Its Photocatalytic Activity

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Abstract

The discharge of synthetic dyes into aquatic environments poses serious environmental challenges due to their toxicity, persistence, and resistance to conventional treatment methods. Photocatalysis using semiconductor nanoparticles has emerged as an effective approach for degrading such pollutants. In this study, zinc oxide (ZnO) nanoparticles were synthesized via pulsed laser ablation in liquid (PLAL), with a focus on the effect of laser scanning area on nanoparticle formation and photocatalytic performance. A high-purity zinc target was ablated in deionized water using a fiber laser at a constant power of 20 W, while the scanning area was varied from 1 x 1 mm² to 5 x 5 mm². The synthesized nanoparticles were characterized using ultraviolet-visible (UV-Vis) spectroscopy and transmission electron microscopy (TEM). The synthesized ZnO nanoparticles exhibited near-hexagonal morphology with an average particle size of 18.10 nm. Photocatalytic activity was evaluated through the degradation of crystal violet dye under UV-A irradiation. The degradation efficiency increased from 84.49% to 97.91% with increasing scanning area, with the highest performance observed at 5 x 5 mm². These findings highlight the critical role of laser scanning area in optimizing ZnO nanoparticle synthesis and enhancing photocatalytic efficiency.

Keywords: zinc oxide nanoparticles, pulsed laser ablation in liquid, laser scanning area, photocatalysis, crystal violet

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1.0 INTRODUCTION

The discharge of synthetic dyes from textile and industrial activities poses a significant threat to aquatic ecosystems and human health due to their toxicity, persistence, and resistance to biodegradation [1]. Conventional treatment approaches such as adsorption, physical separation, and biological degradation are often ineffective for the removal of stable organic dyes due to their complex molecular structures and high chemical stability [2]. As a result, advanced oxidation processes (AOPs) have gained increasing attention, particularly photocatalysis, which utilizes semiconductor materials to generate reactive oxygen species capable of degrading organic contaminants into harmless end products such as carbon dioxide and water [3]. Photocatalytic oxidation has been widely recognized as an efficient route for complete mineralization of organic pollutants in water [4].

Among various semiconductor photocatalysts, zinc oxide (ZnO) nanoparticles have emerged as a promising material due to their excellent optical and electronic properties, including a wide bandgap of 3.37 eV, high electron mobility, and high exciton binding energy of 60 meV [5]. These characteristics enable efficient generation and separation of electron-hole pairs under UV irradiation, enhancing photocatalytic efficiency. Compared to other materials such as titanium dioxide (TiO_2), ZnO often demonstrates comparable or superior photocatalytic performance while offering advantages in terms of cost, abundance, and environmental compatibility [6]. ZnO remains one of the most widely studied semiconductor materials due to its stability, non-toxicity, and multifunctionality [7,8]. The excellent optical and electronic properties of ZnO have made it highly attractive for photocatalytic and environmental applications [9,10]. The synthesis method plays a critical role in determining the physicochemical properties of ZnO nanoparticles, which directly influence their photocatalytic performance. Pulsed Laser Ablation in Liquid (PLAL) has emerged as a green technique for producing high-purity, surfactant-free nanoparticles [11]. Laser ablation in liquid has also been reported as an effective route for producing highly pure oxide nanoparticles without chemical additives [12]. Unlike conventional chemical synthesis methods, PLAL eliminates the need for reducing agents or stabilizers, thereby preserving active surface sites and minimizing contamination. Additionally, PLAL enables precise control over nanoparticle size, morphology, and defect structure through adjustment of laser parameters, leading to enhanced catalytic activity [13].

Previous studies have demonstrated that laser parameters such as laser power, wavelength, and pulse energy significantly influence nanoparticle formation by affecting plasma dynamics, energy distribution, and ablation efficiency. For instance, Ismail et al. reported that ZnO nanoparticles synthesized via pulsed laser ablation using a Nd:YAG laser with wavelengths of 1064 and 532 nm and a pulse duration of 10 ns, at varying laser fluences in the range of 8.7-39.6 J/cm², exhibited changes in optical absorption and band gap energy, demonstrating that increasing laser fluence significantly influences nanoparticle properties [14]. Flemban et al. reported that ZnO nanoparticles synthesized via pulsed laser ablation using a Q-switched Nd:YAG laser (1064 nm, 10 ns) at varying pulse energies of 70, 90, and 130 mJ, corresponding to laser fluences of 18.2, 23.3, and 33.7 J/mm², exhibited significant changes in nanoparticle characteristics, confirming that laser energy strongly influences the structural and optical properties of ZnO nanoparticles [15]. However, while factors such as particle size, composition, and irradiation conditions have been widely investigated, the effect of laser scanning area during PLAL on photocatalytic dye degradation applications remain relatively unexplored. The scanning area governs the spatial distribution of laser energy on the target surface, which directly affects ablation rate, plasma characteristics, and nanoparticle yield. In this study, ZnO nanoparticles were synthesized via PLAL using different laser scanning areas, namely 1 x 1 mm², 2 x 2 mm², 3 x 3 mm², 4 x 4 mm², and 5 x 5 mm². The influence of scanning area on nanoparticle formation and photocatalytic performance was investigated through the degradation of crystal violet dye under UV-A irradiation. This work aims to evaluate the influence of laser scanning area on the formation of ZnO nanoparticles synthesized via PLAL and its impact on their photocatalytic activity.

2.0 EXPERIMENTAL

The ZnO nanoparticles were synthesized via the PLAL technique using a high-purity zinc (Zn) plate with 99.99% purity and dimensions of 0.25 mm x 20 mm x 100 mm (thickness x width x length) as the target material. A pulsed fiber laser (Tenfotai cube 10) was used as the laser source with an average power of 20 W and a wavelength of 1064 nm. The laser beam was directed and scanned across the Zn plate surface using a galvo mirror system to create different scanning sizes during the laser ablation process. Prior to the PLAL process, the Zn plate was mechanically polished using fine sandpaper to remove surface oxides and contaminants. The polished plate was then ultrasonically cleaned in ethanol for 10 minutes to eliminate residual impurities, followed by thorough rinsing with deionized water (DIW) and drying at room temperature. This cleaning procedure ensured a consistent and contaminant-free surface for effective laser ablation.

For the PLAL process, the cleaned Zn plate was immersed in a cuvette containing 5 mL of DIW, as shown in Figure 1. A raster scanning pattern was employed during laser irradiation to achieve homogeneous ablation across the target surface. A pre-ablation step was performed by scanning the entire target surface once at a laser power of 20 W to further remove any remaining surface impurities and stabilize the ablation conditions. To investigate the influence of laser scanning area on nanoparticle formation, five different scanning sizes were applied, namely 1 x 1 mm², 2 x 2 mm², 3 x 3 mm², 4 x 4 mm², and 5 x 5 mm² during PLAL process. An average power of 20 W, a frequency of 20 kHz, and a pulse width of 100 ns were maintained as constant parameters throughout the experiments. The ablation time for each experiment was fixed at 10 seconds, with a scanning speed of 1000 mm/s. This resulted in repeated scans for the scanning areas ranging from 1 x 1 mm² to 4 x 4 mm², whereas the 5 x 5 mm² scanning size received only a single scan. For each experiment, approximately 10 mL of ZnO nanoparticle colloidal suspension was obtained. The synthesized colloids were subsequently sonicated in an ultrasonic bath for 10 minutes to ensure uniform dispersion, reduce agglomeration, and enhance colloidal stability.

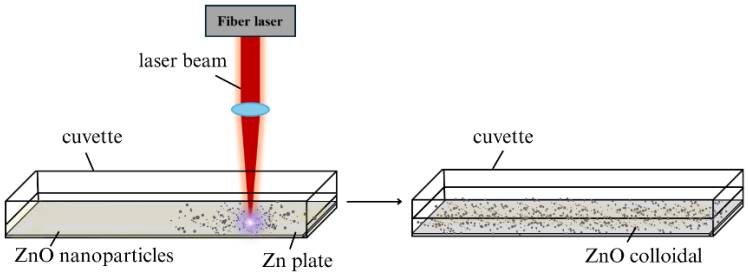


Figure 1 : Experimental setup of the PLAL process for ZnO nanoparticle synthesis

The synthesized ZnO nanoparticles were characterized to evaluate their morphological and structural properties. Transmission electron microscopy (TEM) analysis was performed on a selected sample, identified as the optimum based on synthesis conditions, to represent the physical and morphological characteristics of the produced nanoparticles. This analysis was used to examine particle morphology, size distribution, and lattice structure. The photocatalytic activity of the synthesized ZnO nanoparticles was evaluated through the degradation of crystal violet (CV) dye under UV-A irradiation. A stock solution of CV dye with a concentration of 10 mg/L was prepared using DIW. For each photocatalytic experiment, 20 mL of the CV solution was mixed with 10 mL of ZnO nanoparticle colloid. Prior to light exposure, the mixture was kept in the dark for 30 minutes to establish adsorption-desorption equilibrium between the dye molecules and the catalyst surface as illustrated in Figure 2.

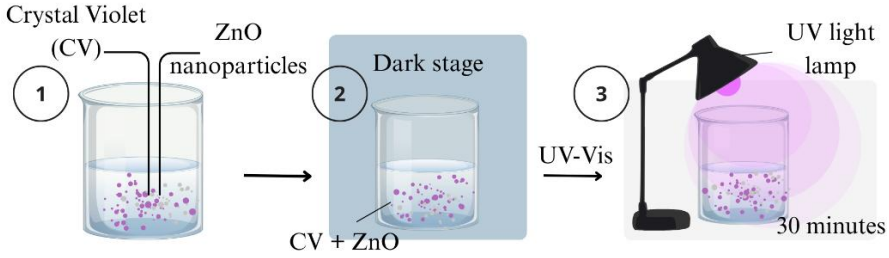


Figure 2: Schematic illustration of the photocatalytic degradation procedure of CV dye under UV-A irradiation

Figure 3 shows photographs of the CV dye solution before (0 min) and after (30 min) photocatalytic treatment using ZnO nanoparticles under UV-A irradiation. At 0 min, the solution exhibits a distinct violet colour, indicating the presence of CV dye molecules. After 30 min of irradiation, the colour intensity decreased significantly, and the solution became almost clear, demonstrating the effective degradation of the dye.



Figure 3 : Photographic images of the CV dye solution at 0 min and after 30 min of photocatalytic degradation using ZnO nanoparticles.

Following adsorption-desorption equilibrium in the dark stage, an initial sample at 0 min was collected to obtain the baseline absorbance. The suspension was then exposed to a UV-A lamp with a wavelength of 368 nm, and a subsequent sample was collected after 30 minutes of irradiation. The absorption changes were recorded using UV-Vis spectroscopy, and the photocatalytic degradation efficiency was determined based on the change in absorbance over time. The degradation efficiency (η) was calculated using the following equation:

$$\eta\ (\%) = \frac{C_0 - C_t}{C_0}\ x\ 100$$

where C_0 is the initial absorbance of the dye after adsorption-desorption equilibrium, and C_t is the absorbance at irradiation time, t.

3.0 RESULTS AND DISCUSSION

This section discusses the results obtained from the characterization and photocatalytic evaluation of the synthesized ZnO nanoparticles. The morphological properties of the nanoparticles were analyzed using TEM, while the photocatalytic performance was assessed through the degradation of crystal violet dye under UV-A irradiation. The influence of laser scanning area on nanoparticle formation and its corresponding effect on photocatalytic activity is discussed.

3.1 Characterization of ZnO nanoparticles

The transmission electron microscopy (TEM) micrograph shown in Figure 4, obtained at a magnification of x100k, shows the morphology of the ZnO nanoparticles synthesized using the 5 x 5 mm² laser scanning area. The observed near-hexagonal morphology is characteristic of the wurtzite ZnO crystal structure and is consistent with previously reported ZnO nanostructures [16]. Figure 5 shows a lattice spacing of 0.25 nm observed in the high-resolution TEM image acquired at x300k magnification, which corresponds to the (101) plane of hexagonal wurtzite ZnO and is consistent with previously reported values [17]. The particle size distribution was determined from TEM micrographs using ImageJ software by calibrating a 100 nm scale bar. A statistically significant number of nanoparticles were measured to obtain the equivalent particle size, as presented in Figure 6. The size distribution histogram shows that the ZnO nanoparticles range between 15 and 25 nm, indicating a relatively narrow size distribution with a mean particle size of 18.10 nm and a standard deviation of 2.71 nm.

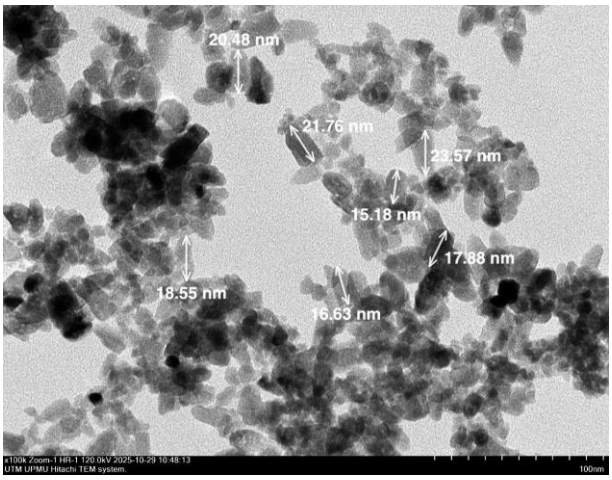


Figure 4 : TEM image of ZnO nanoparticles showing particle morphology at x100k magnification

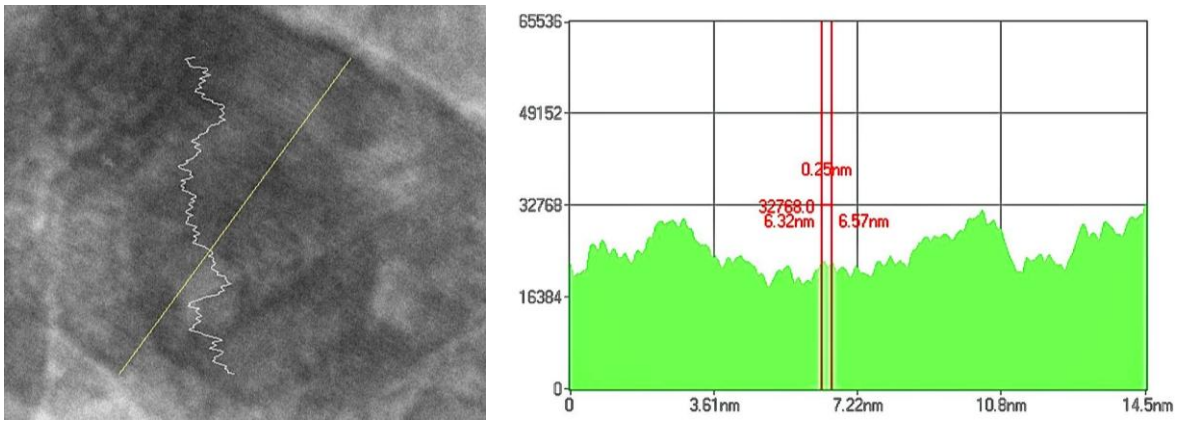


Figure 5 : TEM image of ZnO nanoparticles at x300k magnification showing their morphology and nanoscale structure

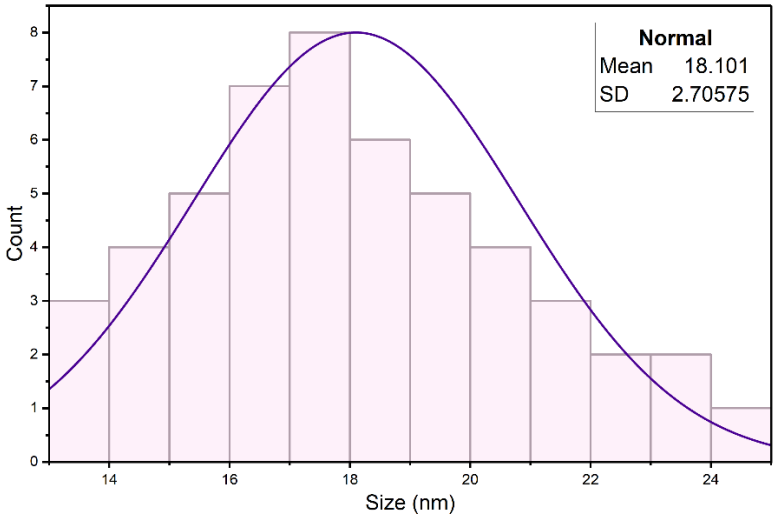
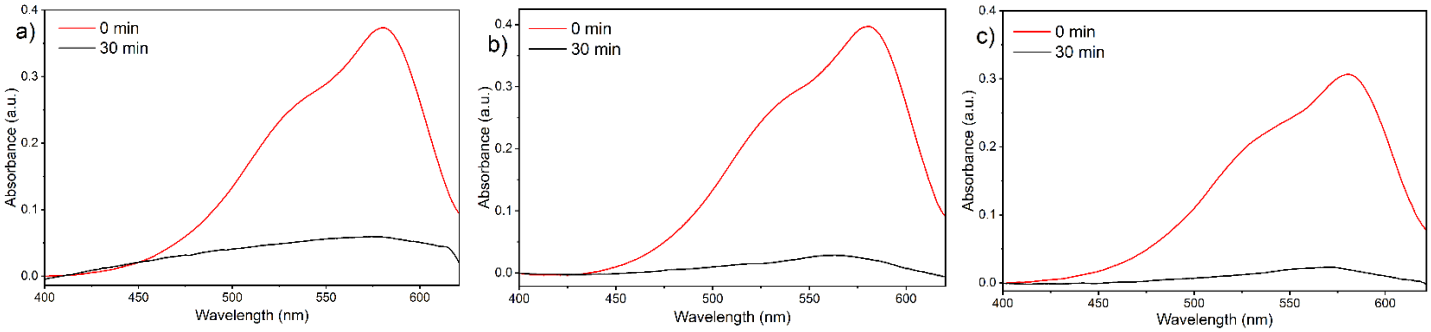


Figure 6 : Particle size distribution histogram of ZnO nanoparticles

3.2 Photodegradation of crystal violet dye using ZnO nanoparticles

At a scanning area of 1 x 1 mm² in Figure 7a, the absorbance decreases from 0.374 to 0.058 a.u. after 30 minutes, corresponding to a degradation efficiency of 84.49%. When the scanning area is increased to 2 x 2 mm² in Figure 7b, the efficiency improves significantly to 92.44%, followed by a further increase to 93.79% at 3 x 3 mm² in Figure 7c. This enhancement can be attributed to a more uniform distribution of laser energy across the target surface, which promotes more effective ablation and increases the concentration of ZnO nanoparticles in the colloidal solution. The higher nanoparticle concentration enhances light absorption and provides more active sites, facilitating the generation of reactive oxygen species responsible for dye degradation [18].

A similar trend is observed at larger scanning areas, where the degradation efficiency increases to 94.94% at 4 x 4 mm² in Figure 7d and reaches a maximum of 97.91% at 5 x 5 mm² in Figure 7e. The higher performance at 5 x 5 mm² indicates that a larger scanning area provides more stable ablation conditions and improves the overall nanoparticle yield. As a result, the increased availability of ZnO nanoparticles enhances light utilization and promotes more effective interaction between the catalyst surface and dye molecules. These factors collectively contribute to improved photocatalytic activity, demonstrating that laser scanning area plays a crucial role in optimizing nanoparticle synthesis and degradation efficiency.



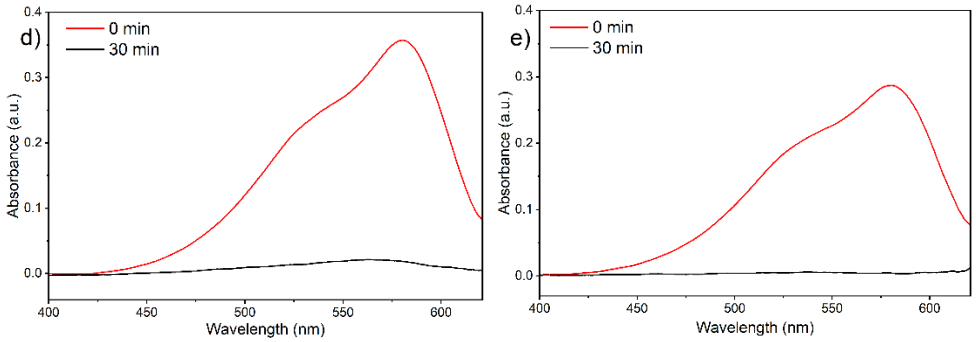


Figure 7 : Photocatalytic degradation of crystal violet (CV) dye using ZnO nanoparticles synthesized at different laser scanning areas: (a) 1 x 1 mm², (b) 2 x 2 mm², (c) 3 x 3 mm², (d) 4 x 4 mm², and (e) 5 x 5 mm²

Figure 8 summarizes the degradation efficiencies of ZnO nanoparticles synthesized at different scanning areas. A clear increasing trend is observed, where the degradation efficiency improves from 84.49% at 1 x 1 mm² to 97.91% at 5 x 5 mm². The photocatalytic degradation of CV dye using ZnO nanoparticles exhibits a clear dependence on the laser scanning area.

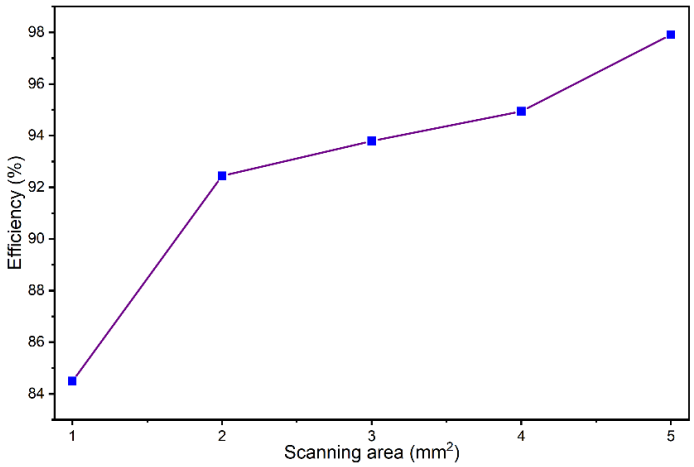


Figure 8 : Comparison of photocatalytic degradation efficiencies of ZnO nanoparticles synthesized at different laser scanning areas

This trend demonstrates that laser scanning area plays a crucial role in determining the photocatalytic performance of ZnO nanoparticles. Unlike laser power, which controls the magnitude of energy input, scanning area influences the spatial distribution of energy and ablation uniformity. In smaller scanning areas, the laser beam repeatedly scans the same region within the fixed 10 second ablation time, causing nanoparticles generated in early scans to accumulate in the colloid and interfere with subsequent laser propagation through absorption and scattering. This reduces the effective laser energy reaching the zinc target, lowering ablation efficiency and nanoparticle yield. In contrast, larger scanning areas involve fewer repeated scans within the same ablation time, reducing nanoparticle accumulation in the beam path, minimizing energy loss, and improving laser reach to the target, which increases nanoparticle formation.

Figure 9 illustrates the photocatalytic degradation mechanism of crystal violet (CV) using ZnO nanoparticles under UV-A irradiation. ZnO nanoparticles absorb photons and generate electron-hole pairs, where electrons in the valence band are excited to the conduction band, leaving positive holes behind. These charge carriers migrate to the nanoparticle surface and participate in redox reactions where the electrons reduce dissolved oxygen to form superoxide radicals ($\bullet O_2^-$), while the holes oxidize water molecules or hydroxide ions to produce hydroxyl radicals (•OH). The generation of these radicals is widely accepted as the dominant pathway in semiconductor photocatalytic degradation processes [19]. These highly reactive species degrade CV dye molecules, breaking their complex structure into smaller intermediate compounds, which are subsequently oxidized and mineralized into CO_2 and H_2O [20]. The higher photocatalytic performance observed at larger scanning areas is attributed to increased nanoparticle concentration and improved light utilization, which enhances the generation of reactive species and overall degradation efficiency.

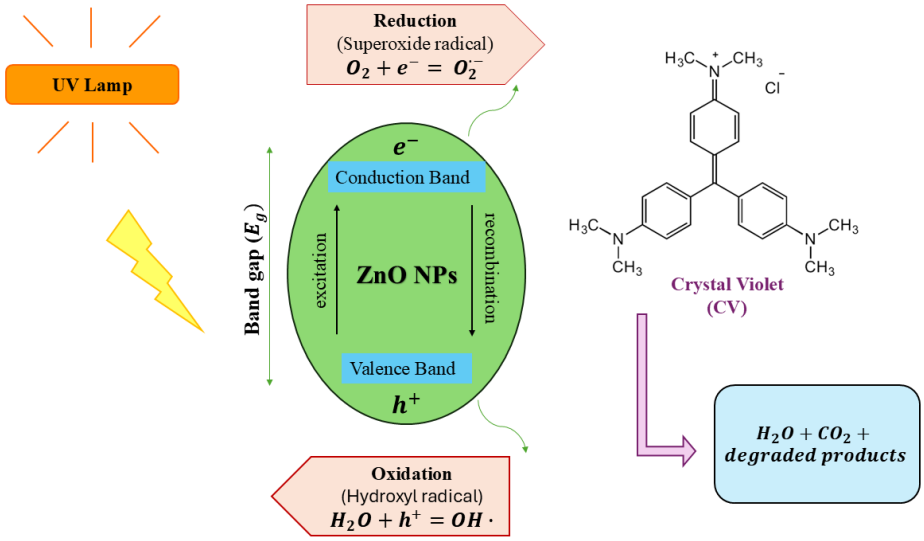


Figure 9 : Schematic illustration of the photocatalytic degradation mechanism of crystal violet dye using ZnO nanoparticles under UV-A irradiation

4.0 CONCLUSION

In this study, ZnO nanoparticles were successfully synthesized via the PLAL technique using different laser scanning areas, and their influence on photocatalytic performance was systematically investigated. The synthesized nanoparticles exhibited near-hexagonal morphology with a relatively narrow size distribution and an average particle size of 18.10 nm. The results demonstrate a clear dependence of photocatalytic efficiency on the laser scanning area, where larger scanning areas significantly improved the degradation performance of CV dye under UV-A irradiation. The degradation efficiency increased progressively from 84.49% at 1 x 1 mm² to 97.91% at 5 x 5 mm², indicating that a larger scanning area provides more uniform energy distribution in subsequent scans and enhances the ablation process. This leads to a higher concentration of ZnO nanoparticles in the colloidal solution, resulting in improved light absorption and increased availability of active sites for photocatalytic reactions. Consequently, more reactive oxygen species are generated, promoting more efficient degradation of dye molecules. Further optimization of laser parameters may enhance nanoparticle characteristics and improve photocatalytic performance in future studies. Thus, this study highlights the importance of laser scanning parameters, particularly scanning area, in controlling nanoparticle synthesis and optimizing photocatalytic performance. The findings provide valuable insight into improving PLAL-based synthesis for efficient environmental applications.

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