

# Comparative Photocatalytic Performance of Gd, Zn, and Ti Metal Oxide Catalysts for Polyethylene Microplastics Removal

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## ABSTRACT

*This study presents a comparative analysis of the photocatalytic degradation efficiency of modified and commercial Gd<sub>2</sub>O<sub>3</sub>, ZnO, and TiO<sub>2</sub> metal oxide catalysts for the removal of polyethylene (PE) microplastics under controlled laboratory conditions. Among the tested catalysts, modified ZnO exhibited the highest degradation efficiency of 78% under optimum conditions: 10 ppm PE concentration, 3-4 g/L catalyst dosage, pH 7, visible light irradiation for 2 hours at 303 K. This was followed by modified Gd<sub>2</sub>O<sub>3</sub> (68%) and modified TiO<sub>2</sub> (58%) under the same conditions. All modified catalysts outperformed their commercial counterparts, while the photolysis control experiment showed less than 5% degradation. The enhanced photocatalytic activity is attributed to improved charge separation, greater generation of reactive oxygen species (ROS), and increased surface adsorption capacity introduced by defect engineering and structural modifications. Negligible degradation in the absence of light confirms the dominance of photocatalytic mechanisms over photolysis or dark reactions. These findings underscore the potential of surface-modified ZnO as a highly effective catalyst for the remediation of microplastic pollution.*

**Keywords :** Gadolinium, zinc, titanium, photocatalytic, polyethylene microplastics, pollution.

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## 1. INTRODUCTION

Microplastic pollution has become a pressing environmental concern, particularly in aquatic ecosystems, where it poses significant risks to biodiversity and human health. Among various microplastic pollutants, polyethylene (PE) microplastics are the most prevalent due to the widespread use of PE in packaging, consumer products, and industrial applications [1,2]. These microplastics are chemically stable, hydrophobic, and highly resistant to biodegradation, allowing them to persist in the environment for extended periods. Once introduced into aquatic systems, PE microplastics can adsorb and transport toxic chemicals, be ingested by aquatic organisms, and enter the food chain, thus threatening both ecological integrity and public health [3].

Conventional methods for removing microplastics such as filtration, coagulation, and sedimentation; mainly rely on physical separation and are often insufficient for complete removal. These techniques can be energy-intensive, generate secondary pollutants, and fail to degrade microplastics into harmless byproducts [4]. Hence, there is an urgent need for innovative, sustainable technologies capable of degrading microplastics at the molecular level [5]. Photocatalysis, which utilizes light energy to produce reactive oxygen species (ROS) for the breakdown of organic contaminants, has emerged as a promising and eco-friendly approach for microplastic remediation [6].

Metal oxide semiconductors, including titanium dioxide (TiO<sub>2</sub>), zinc oxide (ZnO), and gadolinium oxide (Gd<sub>2</sub>O<sub>3</sub>), have been widely investigated as photocatalysts due to their chemical stability, low toxicity, and ROS-generating capabilities under light irradiation [7-9]. TiO<sub>2</sub> is the most extensively studied, though its wide bandgap restricts its activity mainly to the ultraviolet (UV) range. ZnO offers a similar bandgap but provides improved light absorption in the UV-visible region. Meanwhile, rare-earth metal oxides like Gd<sub>2</sub>O<sub>3</sub>, although less explored, possess distinctive electronic structures; particularly partially filled 4f orbitals that promote efficient charge separation and prolong the lifetime of photogenerated electron-hole pairs [10,11].

Recent advancements have shown that surface modification, doping, and defect engineering can significantly enhance the photocatalytic performance of these metal oxides [7,12,13]. Such modifications aim to introduce oxygen vacancies, tune the band structure, and improve pollutant adsorption, thereby increasing ROS production. However, comparative studies evaluating the photocatalytic performance of different modified metal oxide catalysts under identical conditions especially for PE microplastic degradation remain limited.

This study addresses this gap by systematically evaluating and comparing the photocatalytic degradation efficiency of modified and commercial  $\text{Gd}_2\text{O}_3$ ,  $\text{ZnO}$ , and  $\text{TiO}_2$  catalysts for PE microplastic removal. The investigation was conducted under controlled laboratory conditions, focusing on key parameters such as light exposure, pH, catalyst dosage, and pollutant concentration to ensure consistent and meaningful comparisons.

The findings offer valuable insights into the performance of rare-earth-based photocatalysts relative to more conventional systems, highlighting the potential of surface-modified  $\text{ZnO}$  and  $\text{Gd}_2\text{O}_3$  in microplastic remediation. This work contributes to the advancement of photocatalytic technologies for environmental sustainability and provides guidance for future catalyst development aimed at addressing the global microplastic crisis.

## 2. MATERIALS AND METHODS

### 2.1 Materials

Gadolinium oxide ( $\text{Gd}_2\text{O}_3$ ), zinc oxide ( $\text{ZnO}$ ), and titanium dioxide ( $\text{TiO}_2$ ) (all from MACKLIN) were employed as the primary metal oxide photocatalysts in this study. Polyethylene (PE) microplastic powder (MACKLIN) served as the model pollutant. Tween 80 (Chemiz) was added to facilitate uniform dispersion of microplastics in aqueous solutions. *N,N*-Dimethylformamide (DMF; ChemAR) was used as a solvent during catalyst modification. Nile Red dye (MACKLIN) was utilized for fluorescence-based quantification of residual microplastics, with dimethyl sulfoxide (DMSO; Merck) acting as a co-solvent for dye dissolution and microplastic detection. The pH of reaction mixtures was adjusted using 0.1 M hydrochloric acid (HCl) and 0.1 M sodium hydroxide (NaOH), both from QReC™. All chemicals were of analytical grade and used without further purification. Deionized water was used for all solution preparations and experimental procedures.

### 2.2 Preparation of catalyst

The modified  $\text{Gd}_2\text{O}_3$ ,  $\text{ZnO}$ , and  $\text{TiO}_2$  catalysts were synthesized via a combined microwave-assisted and impregnation method. For each metal oxide, the precursor powder was suspended in *N,N*-Dimethylformamide (DMF), selected for its chemical stability and effective dispersing properties. The suspension was subjected to microwave irradiation at 450 W for 15 minutes to promote particle activation and uniform energy distribution. This was followed by ultrasonication for 15 minutes to ensure homogeneous dispersion of the metal oxide particles within the solvent. The resulting suspension was gently heated at 60–80 °C under continuous stirring to evaporate excess DMF, forming a paste-like material. This paste was then dried at 105 °C for 24 hours to remove residual solvent and moisture. The dried product was subsequently calcined at 550 °C for 3 hours in air, using a controlled heating rate of 5 °C/min, to enhance crystallinity and eliminate remaining organic residues. The modified catalysts were allowed to cool naturally to room temperature and stored in airtight containers until further use [14,15].

### 2.3 Preparation of microplastics

Polyethylene (PE) microplastics were selected for this study due to their widespread use, environmental persistence, and unique physicochemical properties that influence degradation mechanisms [2]. For spectrophotometric monitoring and visual tracking, the microplastics were stained with Nile Red, a lipophilic fluorescent dye known for its strong affinity to hydrophobic polymer surfaces [16]. A working dye solution (0.005 g/L) was prepared by a tenfold dilution of a 0.05 g/L Nile Red stock solution in acetone using *n*-hexane as the diluent. Approximately 200 mg of PE microplastics were immersed in 15 mL of the dye solution and incubated at 60 °C for 2 hours. A visible pink-to-violet coloration indicated successful dye adsorption onto the polymer surface. To enhance dispersion in aqueous media, Polysorbate 80 (TWEEN 80) was added at a concentration of 100 mg/L [17]. This non-ionic surfactant reduced

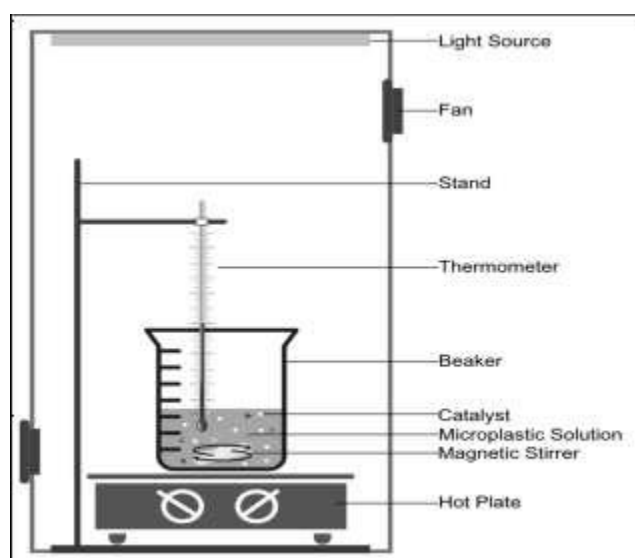
interfacial tension, thereby improving the suspension of the low-density PE particles in water. The stained and surfactant-stabilized microplastics were subsequently diluted to final concentrations of 10, 20, 30, 40, 50, 70, and 100 ppm representing contamination levels commonly detected in environmental samples. This standardized preparation enabled reproducible and environmentally relevant conditions for photocatalytic degradation experiments under visible light irradiation.

#### 2.4 Photocatalytic reaction testing

The photocatalytic activity of the modified  $\text{Gd}_2\text{O}_3$ ,  $\text{ZnO}$ , and  $\text{TiO}_2$  catalysts was assessed based on their ability to degrade polyethylene (PE) microplastics. In each experiment, a specific amount of catalyst (corresponding to 3 g/L or 4 g/L, depending on the experimental condition) was dispersed in 50 mL of a 10 ppm PE microplastic suspension. Tween 80 was added as needed to facilitate uniform dispersion of microplastics. Prior to irradiation, the suspension was magnetically stirred in the dark for 2 hours to establish adsorption–desorption equilibrium. Following equilibration, the system was exposed to light using a 300 W mercury vapor fluorescent lamp (or an equivalent visible/UV light source) positioned approximately 15 cm above the solution surface. The reaction temperature was maintained at 303 K throughout the experiment. At predetermined time intervals, aliquots were withdrawn from the reaction mixture, filtered to remove catalyst particles, and analyzed using a UV–Visible spectrophotometer (Thomas Edison LSS-U2900 Double Beam). Absorbance was measured at 243 nm to quantify the residual microplastic concentration. The degradation efficiency (%) was calculated to evaluate the photocatalytic performance, and to examine the effects of catalyst type, light conditions, and surface modification.

#### 2.5 Simple batch photoreactor system

Photocatalytic experiments were carried out in a custom-designed batch photoreactor system. Illumination was provided by a Philips Lifemax TLD 18W/54 fluorescent lamp (13,000-hour lifespan; 6200 K), emitting in the 280–315 nm wavelength range. The lamp was positioned approximately 15 cm above the reaction flask, as illustrated in Fig. 1. Reactions were conducted in 50 mL Pyrex conical flasks placed within a light-tight chamber lined with aluminum foil to enhance light reflection and ensure uniform irradiation. Continuous stirring was maintained using a magnetic stirrer to achieve consistent catalyst dispersion throughout the reaction. The temperature within the reactor was regularly monitored to ensure stable and uniform thermal conditions during the photocatalytic tests [12,18].



**Fig. 1.** Photodegradation Batch Reactor System

#### 2.6 Determination of microplastic concentration

The concentration of polyethylene (PE) microplastics was determined using a UV–Visible spectrophotometer (Thomas Edison LSS-U2900 Double Beam) by measuring the absorbance at 243 nm, corresponding to the characteristic peak of the TWEEN 80/PE complex. At predetermined time intervals, 1.5 mL aliquots were withdrawn from the reaction mixture and centrifuged to remove residual catalyst

particles. The photocatalytic degradation efficiency (%) was calculated using the following equation:

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

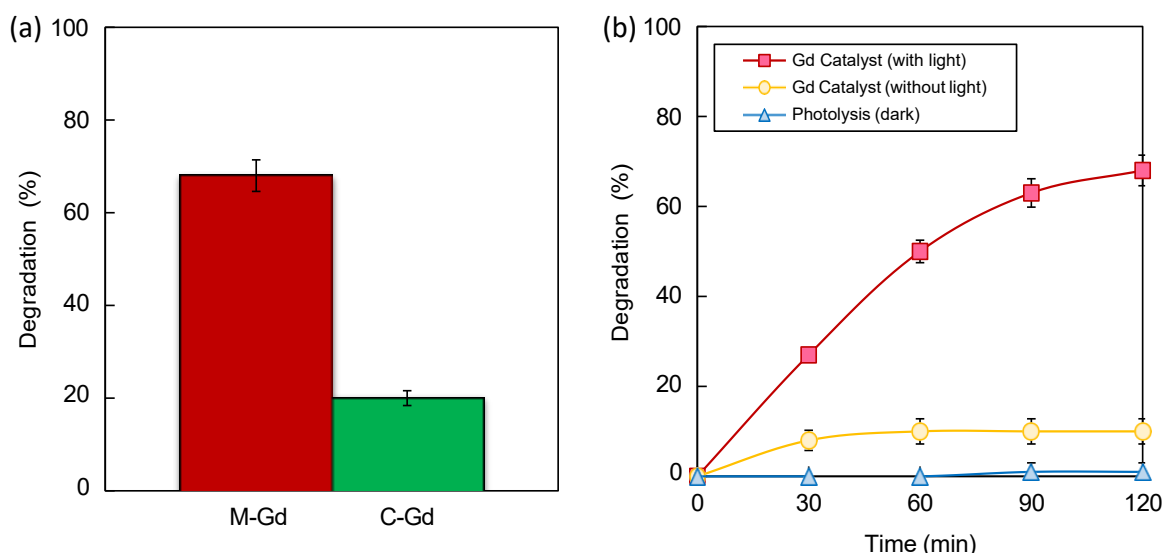
(1)

where  $C_0$  signifies the initial concentration and  $C_t$  represents a changing concentration.

### 3. RESULTS AND DISCUSSION

#### 3.1 Photocatalytic performance of $\text{Gd}_2\text{O}_3$

The photocatalytic activity of gadolinium oxide ( $\text{Gd}_2\text{O}_3$ ) in degrading polyethylene (PE) microplastics was evaluated under controlled experimental conditions: pH 7, catalyst dosage of 3 g/L, PE concentration of 10 ppm, temperature of 303 K, and a reaction time of 2 hours. Both modified  $\text{Gd}_2\text{O}_3$  (M-Gd) and commercial  $\text{Gd}_2\text{O}_3$  (C-Gd) were assessed. The results indicate that M-Gd achieved a significantly higher degradation efficiency (68%) under visible light irradiation, compared to only 20% for C-Gd (Fig. 2a). In the absence of light, both catalysts exhibited minimal degradation activity (approximately 10%), while the photolysis control (light without catalyst) accounted for just 1%, confirming the negligible effect of direct photodegradation (Fig. 2b). The time-dependent degradation profile showed a rapid initial phase within the first 60 minutes, followed by a plateau, likely due to the saturation of active sites or the formation of low-molecular-weight PE fragments that are more resistant to further oxidation.



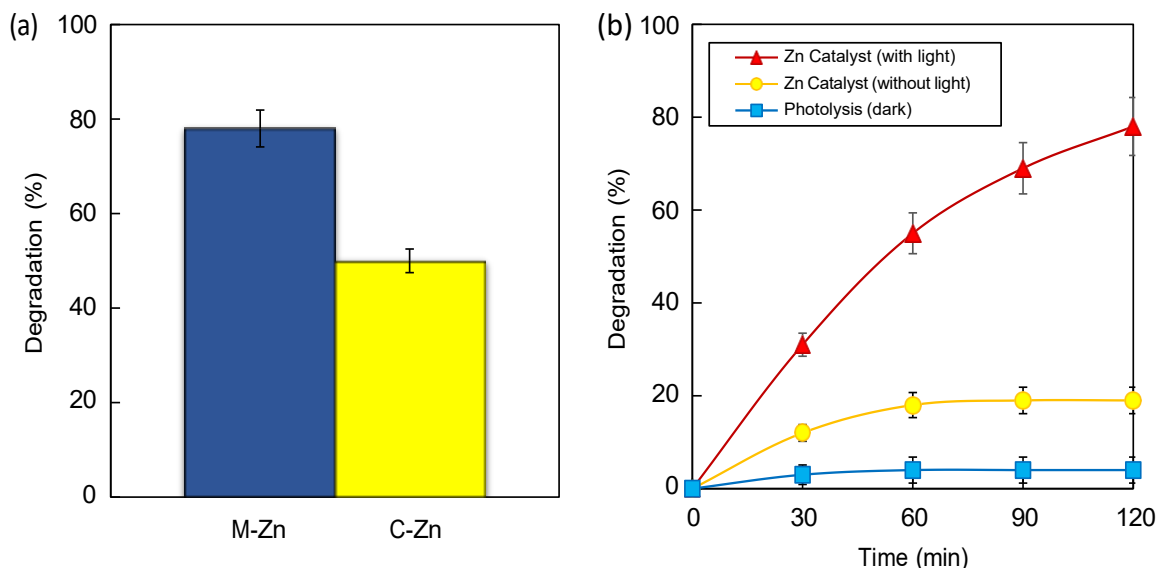
**Fig. 2.** Degradation efficiency of polyethylene microplastics: (a) comparison between modified Gadolinium (M-Gd) and commercial Gadolinium (C-Gd) catalysts, and (b) effect of lighting conditions (light, no light, and photolysis control) [pH = 7; W = 3 g/L; C = 10 ppm; t = 2 h; T = 303 K]

The enhanced performance of M-Gd is attributed to the presence of  $\text{Gd}^{*+}$  ions and the structural improvements induced by the modification process [7,10,19]. Gadolinium's partially filled 4f orbitals effectively trap photogenerated electrons, thereby reducing the recombination rate of electron-hole pairs and increasing the yield of reactive oxygen species (ROS), including hydroxyl radicals ( $\bullet\text{OH}$ ) and superoxide radicals ( $\bullet\text{O}_2^-$ ), which facilitate the oxidative cleavage of PE polymer chains [7,10,19,20]. Furthermore, the modification is believed to have introduced oxygen vacancies and improved surface morphology, thereby increasing the catalyst's surface area and adsorption capacity for microplastics [7,10,19,20]. The low efficiency observed in the photolysis and dark conditions underscores the necessity of photocatalytic activation for effective PE degradation.

#### 3.2 Photocatalytic performance of ZnO

Figure 3 illustrates the photocatalytic performance of zinc oxide (ZnO) catalysts in degrading polyethylene (PE) microplastics. Experiments were conducted under acidic conditions (pH 3), using a catalyst dosage

of 3 g/L, PE concentration of 10 ppm, a reaction time of 2 hours, and a temperature of 303 K. The evaluation compares modified ZnO (M-Zn) and commercial ZnO (C-Zn) under various lighting conditions. Under visible light irradiation, M-Zn exhibited the highest degradation efficiency at 78%, significantly outperforming C-Zn, which achieved 50% (Fig. 3a). In the absence of light, degradation dropped to 19%, while photolysis alone resulted in only 4% degradation (Fig. 3b). The degradation rate was most rapid during the first 60 minutes, followed by a plateau phase likely caused by reduced availability of reactive oxygen species (ROS) or limited accessibility of the remaining microplastics.



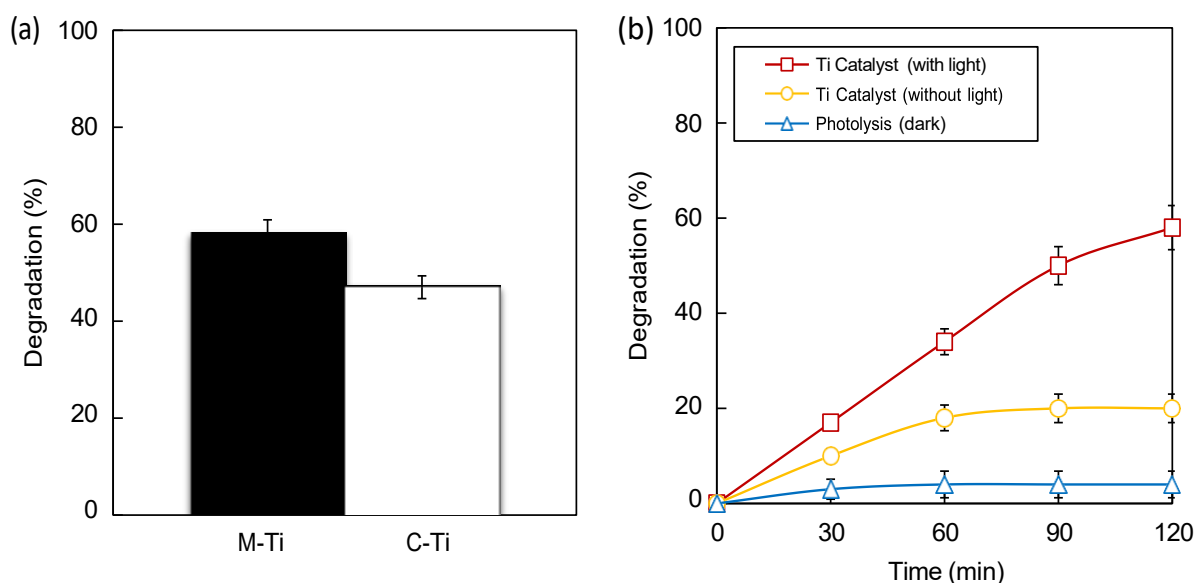
**Fig. 3.** Degradation efficiency of polyethylene microplastics: (a) comparison between modified ZnO (M-Zn) and commercial ZnO (C-Zn) catalysts, and (b) effect of lighting conditions (light, no light, and photolysis control) [pH = 3; W = 3 g/L; C = 10 ppm; t = 2 h; T = 303 K]

ZnO's strong photocatalytic performance is attributed to its effective generation of ROS under visible light [8,12,21]. The enhanced activity of M-Zn is likely due to defect engineering specifically, the introduction of oxygen vacancies and structural defects that function as electron traps, improving charge separation and boosting ROS production [22,23]. Additionally, the acidic pH may have facilitated proton-coupled electron transfer reactions and increased surface interactions between the catalyst and PE microplastics, thereby accelerating degradation [24]. The superior performance of M-Zn over C-Zn highlights the effectiveness of surface modification strategies in enhancing both photocatalytic reactivity and pollutant adsorption. Moreover, ZnO's relatively higher degradation efficiency under no-light conditions, compared to  $\text{Gd}_2\text{O}_3$ , may be attributed to the presence of residual surface-bound redox-active oxygen species capable of sustaining slow oxidative processes even in the absence of direct illumination.

### 3.3 Photocatalytic performance of $\text{TiO}_2$

The photocatalytic efficiency of titanium dioxide ( $\text{TiO}_2$ ) catalysts in degrading polyethylene (PE) microplastics is presented in Figure 4. Experiments were conducted at pH 3, using a catalyst dosage of 4 g/L, PE concentration of 10 ppm, reaction time of 2 hours, and temperature of 303 K. Both modified  $\text{TiO}_2$  (M-Ti) and commercial  $\text{TiO}_2$  (C-Ti) were tested under light, no-light, and photolysis conditions. Under light irradiation, M-Ti achieved a degradation efficiency of 58%, slightly higher than C-Ti at 47% (Fig. 4a). In the absence of light, degradation dropped to 20%, while photolysis alone accounted for only 4%. The time-dependent degradation curve (Fig. 4b) shows a steady but slower degradation trend compared to ZnO.  $\text{TiO}_2$ 's photocatalytic activity is well established and primarily attributed to its ability to generate reactive oxygen species (ROS) upon UV light excitation. However, its relatively wide bandgap ( $\sim 3.2$  eV) restricts light absorption to the UV region, resulting in lower efficiency under visible light compared to ZnO [8,9,22]. The modification process may have introduced surface defects or slightly narrowed the bandgap, but the improvement in performance was modest relative to ZnO [8,9,22,25]. The

20% degradation observed without light may be due to the presence of chemisorbed oxygen species or enhanced surface interactions under acidic conditions. Nevertheless, the process remains predominantly reliant on photocatalytic ROS generation. The slower degradation kinetics further reflect  $\text{TiO}_2$ 's comparatively lower ROS yield under the tested conditions.



**Fig. 4.** Degradation efficiency of polyethylene microplastics: (a) comparison between modified  $\text{TiO}_2$  (M-Ti) and commercial  $\text{TiO}_2$  (C-Ti) catalysts, and (b) effect of lighting conditions (light, no light, and dark/photolysis) [pH = 3; W = 4 g/L; C = 10 ppm; t = 2 h; T = 303 K]

#### 4. CONCLUSION

This study systematically compared the photocatalytic degradation performance of modified and commercial  $\text{Gd}_2\text{O}_3$ ,  $\text{ZnO}$ , and  $\text{TiO}_2$  catalysts for the removal of polyethylene (PE) microplastics under controlled conditions. Among the tested materials, modified  $\text{ZnO}$  exhibited the highest degradation efficiency (78%) under light irradiation, followed by modified  $\text{Gd}_2\text{O}_3$  (68%) and modified  $\text{TiO}_2$  (58%). In all cases, the modified catalysts significantly outperformed their commercial counterparts, confirming that surface modification and defect engineering are critical for enhancing photocatalytic activity by improving charge separation and promoting reactive oxygen species (ROS) generation. The results also emphasized the essential role of light in activating photocatalysts. Degradation efficiencies dropped markedly in the absence of light, while photolysis alone was negligible. This highlights the predominance of ROS-driven photocatalytic mechanisms over non-photocatalytic or direct photolytic pathways. Notably,  $\text{Gd}_2\text{O}_3$  demonstrated competitive performance, suggesting its potential as a viable alternative or complementary material to traditional semiconductors for microplastic degradation. The comparative data generated offer valuable insights into catalyst design and selection for environmental remediation targeting microplastics. Modified  $\text{ZnO}$ , with its superior efficiency and cost-effectiveness, emerges as a strong candidate for further development and scale-up. Meanwhile, the unique electronic properties of  $\text{Gd}_2\text{O}_3$  merit further investigation, particularly in hybrid or composite systems where synergistic interactions could enhance photocatalytic performance. Future studies should focus on optimizing catalyst formulations, developing visible-light-active systems to improve energy efficiency, and evaluating catalyst stability and reusability under realistic environmental conditions. Additionally, mechanistic investigations involving intermediate identification and toxicity assessments are crucial to ensure the environmental safety of photocatalytic degradation products. Overall, this research provides a foundation for advancing practical and sustainable approaches to mitigate microplastic pollution in aquatic environments.

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