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Innovative Zn-Doped TiO₂ Catalysts for Adsorptive–Photocatalytic Removal of Polyethylene Microplastics

Mohammad Luqman Ismail¹, Norzahir Sapawe^{1,*}, Muhammad Farhan Hanafi², Diyana Faziha Mohamad¹, Norezatul Shahirah Ahmad Zamanhuri¹, Mohammed Danish³, Siti Fatimah Ibrahim⁴

- ¹ Universiti Kuala Lumpur, Branch Campus Malaysian Institute of Chemical and Bioengineering Technology (UniKL MICET), Lot 1988 Vendor City, Taboh Nanning, 78000 Alor Gajah, Melaka, Malaysia
² Department of Chemical Engineering and Energy Sustainability, Faculty of Engineering, Universiti Malaysia Sarawak (UNIMAS), 94300 Kota Samarahan, Sarawak, Malaysia
³ Department of Chemistry, Faculty of Science, Islamic University of Madinah (IUM), Abo Bakr Al Siddiq Road, Al Jamiah District, Madinah 42351, Kingdom of Saudi Arabia
⁴ School of Chemical and Process Engineering, University of Leeds, LS2 9JT Leeds, United Kingdom

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ABSTRACT

Polyethylene (PE) microplastics persist in aquatic systems, posing ecological and health concerns due to their high resistance to natural degradation. This work explores the application of microwave-assisted zinc-doped titanium dioxide (Zn–TiO₂) catalysts for the combined adsorption and photocatalytic removal of PE microplastics under visible light. Catalysts were synthesized through microwave modification and assessed across different pH levels, catalyst dosages, and initial PE concentrations. The highest photocatalytic efficiency (85%) was obtained under acidic conditions (pH 3) at an optimal dosage of 3 g/L with 10 mg/L of PE. Zn–TiO₂ exhibited superior performance compared to pristine TiO₂ and ZnO, demonstrating enhanced adsorption and photocatalytic activity attributed to the synergistic effect of Zn incorporation, which improved light utilization and reduced electron–hole recombination. Reusability tests confirmed good stability, with 70% efficiency retained after five cycles. These findings establish microwave-modified Zn–TiO₂ as a promising, durable, and sustainable photocatalyst for mitigating microplastic pollution in aqueous environments.

1. Introduction

The growing prevalence of microplastic pollution has become a critical environmental concern, threatening both aquatic and terrestrial ecosystems. Defined as plastic fragments smaller than 5 mm, microplastics are highly persistent and capable of adsorbing toxic compounds, which complicates their removal from the environment. Conventional treatment methods such as filtration, coagulation, and thermal processing are often costly, inefficient, or environmentally unsustainable, highlighting the urgent demand for cleaner and more effective alternatives [1].

* Corresponding author.

E-mail address: norzahir@unikl.edu.my

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Polyethylene (PE), polypropylene (PP), and polystyrene (PS) are among the most dominant types of microplastics, largely due to their extensive use in packaging, household products, and industrial applications. In Malaysia, PE alone is estimated to account for 45–60% of microplastic waste, followed by PP (20–35%) and PS (5–15%) [2]. The persistence of these polymers in aquatic systems poses serious threats to marine organisms, promotes bioaccumulation, and ultimately raises concerns regarding food security and human health [3].

Photocatalysis has emerged as a promising strategy for degrading such persistent contaminants. Titanium dioxide (TiO_2) is widely recognized as an effective photocatalyst owing to its strong oxidative capacity, low cost, chemical stability, and non-toxic nature. When irradiated with ultraviolet (UV) light, TiO_2 generates reactive oxygen species (ROS) that can break down long polymer chains into smaller, less hazardous byproducts [4]. Nonetheless, its practical application is constrained by a relatively large bandgap (~ 3.2 eV), which restricts activation primarily to UV light, and by the rapid recombination of photo-induced charge carriers, which diminishes its efficiency [5].

To overcome these limitations, doping TiO_2 with transition metals such as zinc (Zn) has been investigated as a means to enhance its photocatalytic performance. Zn doping can reduce the bandgap, extend photoactivity into the visible spectrum, and introduce surface defects that promote the adsorption of hydrophobic pollutants. Moreover, microwave-assisted synthesis provides a rapid and energy-efficient route for tuning the structural and surface properties of TiO_2 nanoparticles, further improving their catalytic efficiency [6].

This study focuses on the adsorptive–photocatalytic removal of polyethylene microplastics using Zn-doped TiO_2 nanocatalysts synthesized via microwave-assisted methods. By optimizing key parameters such as pH, catalyst dosage, and initial microplastic concentration, as well as evaluating catalyst reusability, the research seeks to contribute to the advancement of efficient and sustainable nanomaterials for large-scale microplastic remediation.

2. Methodology

2.1 Chemicals and Materials

Titanium dioxide (TiO_2 , purity $\geq 98\%$, Acros Organics) was employed as the base photocatalyst, while zinc oxide (ZnO, ChemAR) served as the dopant precursor for the synthesis of Zn-doped TiO_2 nanocatalysts. Polyethylene (PE) microplastic powder and Nile Red dye, utilized for degradation studies and fluorescence visualization, were procured from MACKLIN. To aid the dispersion of hydrophobic PE in aqueous media, Tween 80 (Chemiz), a nonionic surfactant, was introduced. N,N-Dimethylformamide (DMF, ChemAR) acted as the solvent during catalyst preparation, while dimethyl sulfoxide (DMSO, Merck) supported additional analytical tests. All chemicals were of analytical grade and used without further purification. Deionized water was used throughout, and pH adjustments were performed with 0.1 M hydrochloric acid (HCl) and sodium hydroxide (NaOH) supplied by QReCTM.

2.2 Synthesis of Zn-Doped TiO_2 Catalyst

Zn-doped TiO_2 was synthesized via a combined microwave-assisted and solid-state doping approach. Predetermined weight ratios of TiO_2 and ZnO were dispersed in N,N-Dimethylformamide (DMF) to achieve the desired Zn doping concentration. The suspension was first subjected to microwave irradiation to activate surfaces and enhance interaction between TiO_2 and ZnO, followed by ultrasonication to promote uniform dispersion of Zn across the TiO_2 lattice. The mixture was then heated at 60–80 °C under stirring to gradually evaporate excess DMF, producing a viscous paste. This

paste was oven-dried at 105 °C for 24 h to remove residual solvent and moisture. The dried precursor was subsequently calcined at 550 °C for 3 h in ambient air at a heating rate of 5 °C/min, enhancing crystallinity, facilitating Zn incorporation, and eliminating organic remnants. The final Zn–TiO₂ nanocatalyst was naturally cooled to room temperature and stored in sealed containers to prevent contamination [7,8].

2.3 Preparation of Polyethylene Microplastics

Polyethylene (PE) was selected as the model microplastic due to its widespread occurrence in plastic waste and environmental persistence [2]. For optical tracking and spectrophotometric detection, PE particles were stained with Nile Red, a hydrophobic dye with strong affinity for nonpolar polymers [9]. A working dye solution (0.005 g/L) was prepared by diluting a 0.05 g/L Nile Red stock (acetone) tenfold in n-hexane. Approximately 200 mg of PE was immersed in 15 mL of the solution and incubated at 60 °C for 2 h, after which the particles exhibited a visible pink-violet coloration, confirming dye adsorption. To stabilize PE dispersions in water, Tween 80 was added at 100 mg/L, reducing interfacial tension and preventing aggregation of the low-density polymer (0.88–0.96 g/cm³). The stained PE microplastics were then diluted into working concentrations of 10, 20, 30, 40, 50, 70, and 100 ppm, reflecting levels commonly detected in environmental matrices [1]. This procedure ensured reproducible and environmentally relevant test conditions for adsorption–photocatalysis experiments.

2.4 Adsorptive–Photocatalytic Degradation Procedure

The performance of Zn-doped TiO₂ was evaluated under dark (adsorption) and illuminated (photocatalysis) conditions. PE suspensions with Zn–TiO₂ were first stirred in darkness to quantify adsorption. For photocatalytic trials, identical suspensions were irradiated with a fluorescent lamp positioned above the reactor. At designated time intervals, aliquots were withdrawn and analyzed. Residual PE concentrations were determined using a Double Beam UV–Vis Spectrophotometer U-2900 (Thomas Edison) within 190–1,100 nm. Absorbance was measured at 234 nm, characteristic of the PE–Tween 80 complex. The decrease in absorbance over time was used to calculate degradation efficiency, allowing comparison of adsorption versus photocatalytic removal.

2.5 Batch Reactor Setup

Photocatalytic studies were conducted in a custom-built batch photoreactor. Illumination was provided by a Philips Lifemax TLD 18W/54 fluorescent lamp (6200 K, 13,000 h lifespan) placed at a fixed distance above the reaction vessel (Fig. 1). All reactions were carried out in Pyrex flasks inside a sealed chamber lined with aluminum foil to maximize light reflection and distribution. Continuous magnetic stirring was maintained to ensure homogenous suspension of microplastics and catalyst. Reactor temperature was monitored throughout to prevent thermal interference with the photocatalytic process. For adsorption experiments, the same setup was used but with complete shielding from light, ensuring a direct comparison of adsorption capacity and photocatalytic performance of the Zn–TiO₂ catalyst [7,10].

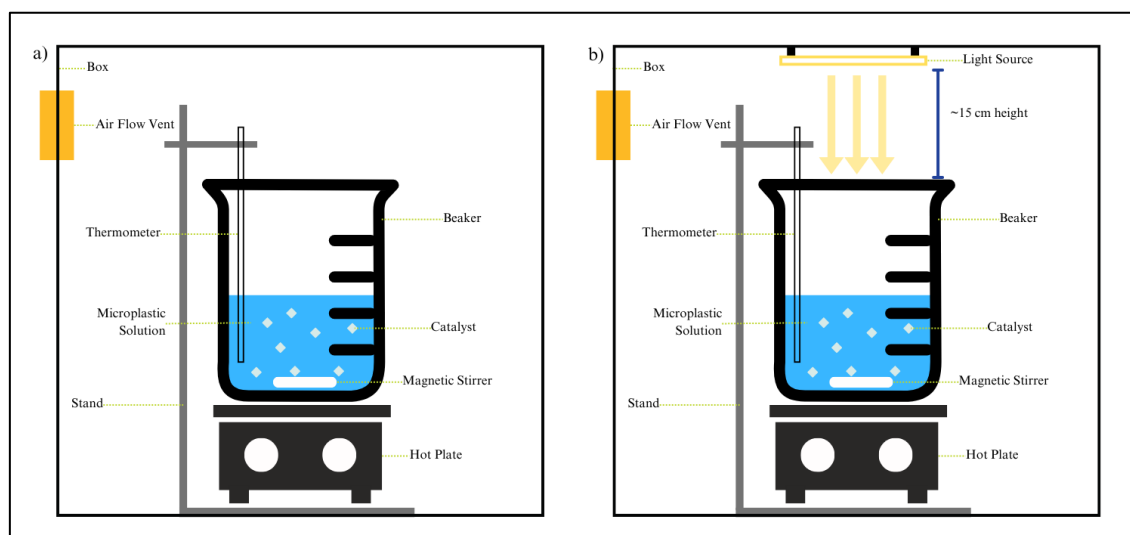


Fig. 1. Schematic of the batch reactor system: (a) adsorption, (b) photocatalytic degradation

2.6 Determination of Polyethylene (PE) Microplastic Concentration

The concentration of polyethylene (PE) microplastics was quantified using a double-beam UV–Vis spectrophotometer (Thomas Edison LSS-U2900), with absorbance recorded at 234 nm, the characteristic wavelength of Nile Red-stained PE particles dispersed in Tween 80. During both adsorption and photocatalytic experiments, 1.5 mL aliquots of the suspension were collected at predetermined intervals and centrifuged to remove catalyst residues prior to analysis. The degradation efficiency of PE microplastics was determined from the decrease in absorbance using the following equation:

$$\text{Degradation (\%)} = \left(\frac{C_0 - C_t}{C_0} \right) \times 100 \quad (1)$$

Where C_0 = initial concentration of PE microplastics (mg/L), and C_t = concentration at time t (mg/L)

This calculation was systematically applied to assess and compare the adsorption and photocatalytic performance of Zn-doped TiO_2 under dark and illuminated conditions.

3. Results and Discussion

3.1 Effect of pH on Adsorption and Photocatalytic Degradation

The removal efficiency of polyethylene (PE) microplastics using Zn-doped TiO_2 was found to be strongly dependent on solution pH. As illustrated in Fig. 2a (adsorption) and Fig. 2b (photocatalysis), both processes exhibited a pH-sensitive behavior, with markedly higher removal under acidic conditions and a gradual decline as the medium shifted towards alkalinity. Experiments were conducted under fixed parameters: catalyst dosage = 3 g/L, PE concentration = 10 mg/L, reaction time = 2 h, and temperature = 303 K.

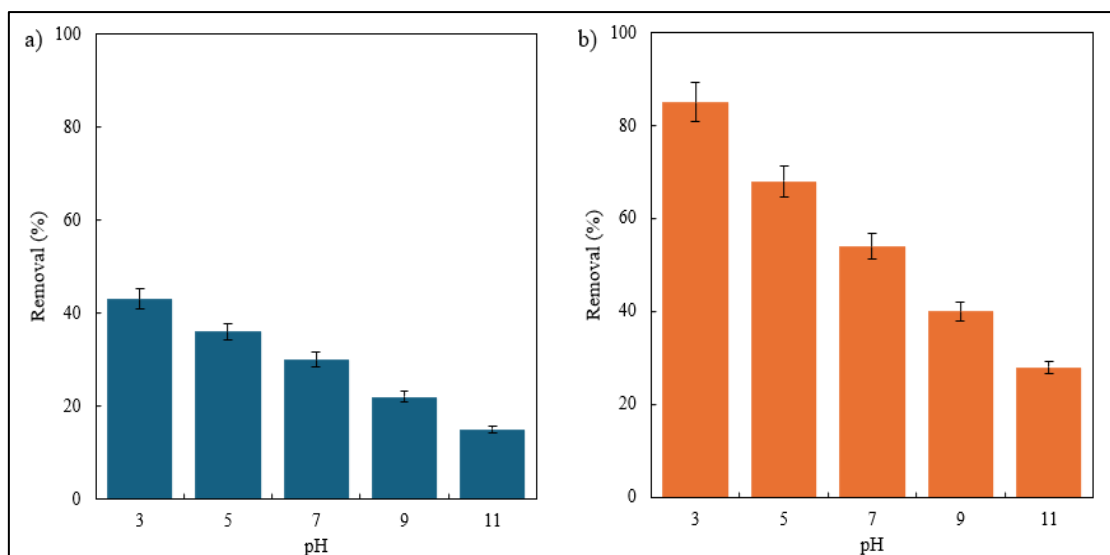


Fig. 2. Effect of pH on polyethylene removal using microwave-synthesized Zn-doped TiO₂ catalyst [W = 3 g/L, C = 10 mg/L, t = 2 h, T = 303 K]: (a) adsorption, (b) photocatalysis

For photocatalytic degradation (Fig. 2b), the highest efficiency was recorded at pH 3, reaching 85%. The removal progressively decreased with increasing pH: 68% at pH 5, 54% at pH 7, 40% at pH 9, and a minimum of 28% at pH 11. This trend highlights the pivotal influence of surface chemistry and reactive oxygen species (ROS) formation particularly hydroxyl radicals ($\bullet\text{OH}$) in driving photocatalytic activity. At acidic pH values (below the isoelectric point of TiO₂, $\sim\text{pH } 6$), the catalyst surface becomes positively charged, enhancing electrostatic interactions with negatively charged intermediates or PE–Tween 80 complexes. These interactions not only favor pollutant adsorption but also facilitate oxidative attack by photo-generated ROS. At neutral pH (7), degradation efficiency declined to 54%, which can be attributed to charge neutrality at the catalyst surface, weakening interactions with PE microplastics. Under alkaline conditions, TiO₂ surfaces develop a negative charge, leading to electrostatic repulsion from negatively charged species present in the suspension [11]. Moreover, elevated hydroxide ion concentrations at pH 9 and 11 can scavenge $\bullet\text{OH}$ radicals, thereby reducing their availability for oxidative degradation.

The adsorption-only experiments (Fig. 2a) exhibited a similar but less pronounced pattern. Maximum adsorption occurred at pH 3 (33%), decreasing to 26% at pH 5, 20% at pH 7, 12% at pH 9, and only 5% at pH 11. These findings demonstrate that adsorption is also governed by surface charge dynamics, though without the additional enhancement provided by photocatalysis. The consistent decline in adsorption with increasing pH indicates diminished affinity between the catalyst and PE microplastics in alkaline media [12]. In summary, both adsorption and photocatalytic removal of PE microplastics were most effective under acidic to mildly acidic conditions (pH 3–5), with photocatalysis consistently achieving higher removal efficiencies. These results emphasize pH as a critical operational factor in optimizing TiO₂-based microplastic remediation. The observed patterns are consistent with previous reports, which also highlighted enhanced photocatalytic activity in acidic environments due to stronger adsorption, improved charge separation, and increased ROS production [13–15].

3.2 Effect of Catalyst Dosage on Adsorption and Photocatalytic Degradation

The effect of Zn-doped TiO₂ dosage on polyethylene (PE) microplastic removal was investigated at pH 3 with fixed conditions: PE concentration = 10 mg/L, reaction time = 2 h, and temperature =

303 K. Catalyst loadings ranging from 1 to 6 g/L were tested for both adsorption and photocatalytic processes, as illustrated in Fig. 3a and Fig. 3b, respectively. For photocatalytic degradation (Fig. 3b), efficiency increased substantially with catalyst dosage up to an optimal threshold. At 1 g/L, degradation reached only 42%, reflecting insufficient active surface sites for light absorption and ROS generation. Increasing the dosage to 2 g/L raised the efficiency to 59%, while the maximum of 85% was achieved at 3 g/L. This enhancement is attributed to the greater availability of photocatalytically active sites, enabling more efficient electron–hole pair generation and ROS production ($\bullet\text{OH}$ and $\text{O}_2\bullet^-$), which drive the oxidative cleavage of polymer chains. Additionally, the expanded surface area facilitated stronger interaction between the catalyst and microplastic particles.

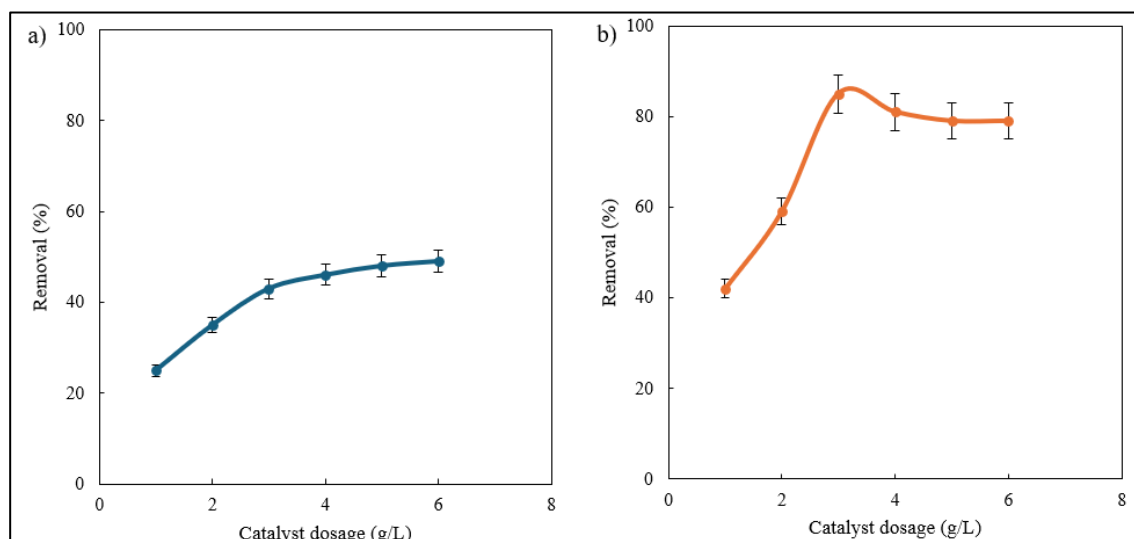


Fig. 3. Effect of catalyst dosage on polyethylene removal using microwave-synthesized Zn-doped TiO_2 catalyst [pH = 3, C = 10 mg/L, t = 2 h, T = 303 K]: (a) adsorption, (b) photocatalysis

Beyond 3 g/L, efficiency declined slightly—81% at 4 g/L and 79% at both 5 and 6 g/L. This plateau and minor reduction can be explained by excess catalyst causing light scattering and shielding effects, which limit photon penetration through the suspension. At higher dosages, particle aggregation may also occur, reducing the effective surface area and the number of accessible active sites. These results indicate the presence of an optimal dosage, here identified as 3 g/L, beyond which further increases provide diminishing or even adverse effects. Adsorption under dark conditions (Fig. 3a) followed a comparable trend, though at lower overall efficiencies. At 1 g/L, adsorption removal was 15%, which gradually increased to 25% (2 g/L) and 33% (3 g/L). Further increases produced modest gains: 36% (4 g/L), 38% (5 g/L), and 39% (6 g/L). The steady improvement is attributed to increased surface area for microplastic binding. Unlike photocatalysis, light scattering is not a limiting factor in adsorption-only systems; thus, removal efficiency depends primarily on surface interactions. Nevertheless, the lower efficiencies compared to photocatalysis confirm the dominant role of light activation and ROS formation in driving microplastic degradation.

Overall, these results highlight the necessity of optimizing catalyst dosage to balance surface area with effective light utilization. While higher catalyst loadings initially promote enhanced removal, excessive amounts lead to photon attenuation and particle agglomeration. For the Zn-doped TiO_2 system, 3 g/L was determined to be the most efficient dosage for PE microplastic degradation. These findings are consistent with prior studies on photocatalytic pollutant removal, which similarly identified an optimum catalyst concentration as essential for maximizing efficiency and ensuring cost-effectiveness in real-world applications [16,17].

3.3 Effect of Initial Polyethylene (PE) Concentration on Adsorption and Photocatalytic Degradation

The influence of the initial polyethylene (PE) microplastic concentration on the performance of Zn-doped TiO₂ was evaluated under controlled operating conditions: pH 3, catalyst dosage = 3 g/L, reaction time = 2 h, and temperature = 303 K. The concentration-dependent behavior for both adsorption and photocatalytic processes, across a range of 10–100 mg/L, is presented in Fig. 4a and Fig. 4b, respectively. Under photocatalytic conditions (Fig. 4b), the degradation efficiency showed a clear inverse correlation with initial PE concentration. At 10 mg/L, the process achieved maximum degradation of 85%, reflecting optimal utilization of active sites and sufficient light penetration. As concentration increased, efficiency decreased progressively to 68% (20 mg/L), 61% (30 mg/L), 50% (50 mg/L), 42% (70 mg/L), and 33% (100 mg/L). This decline is primarily due to the disproportionate pollutant-to-catalyst ratio, which results in surface saturation and restricted interaction of ROS with microplastic particles.

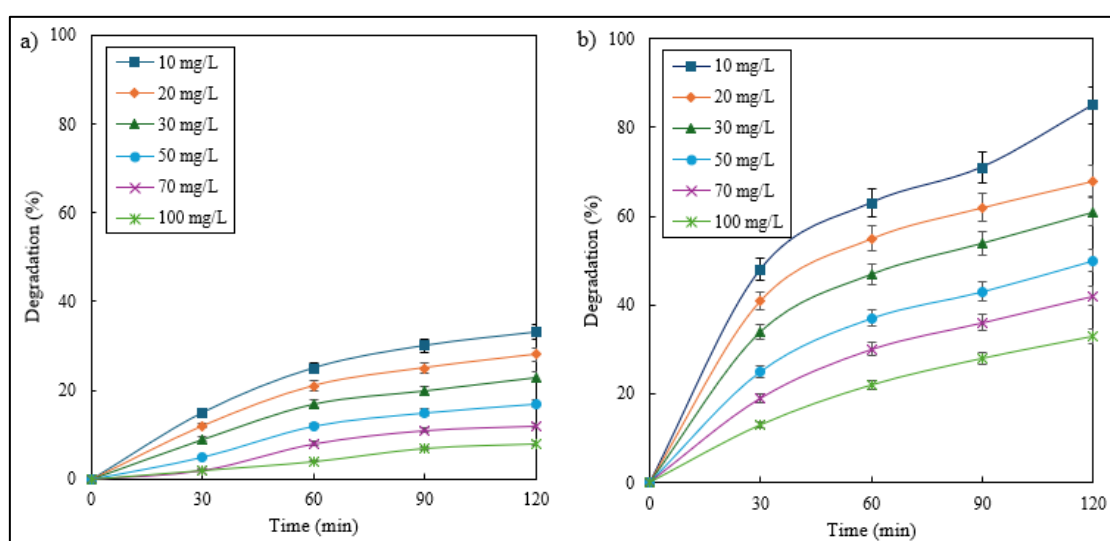


Fig. 4. Effect of initial polyethylene microplastic concentration on PE removal using microwave-modified Zn-doped TiO₂ catalyst [pH = 3, W = 3 g/L, t = 2 h, T = 303 K]: (a) Adsorption, (b) Photocatalytic

Furthermore, higher PE concentrations increase turbidity, limiting light penetration and thereby reducing TiO₂ activation. This weakens photon absorption, suppressing electron–hole pair formation and the subsequent production of reactive species such as hydroxyl radicals ($\bullet\text{OH}$). In addition, excess PE may scavenge ROS competitively, further hindering photocatalytic efficiency. The adsorption-only pathway (Fig. 4a) displayed the same inverse relationship, though at lower efficiency levels. At 10 mg/L, adsorption reached 33%, before decreasing to 28% (20 mg/L), 23% (30 mg/L), 17% (50 mg/L), 12% (70 mg/L), and 8% (100 mg/L). The reduced performance at higher concentrations is attributed to limited active site availability on Zn-doped TiO₂, progressive site saturation, and microplastic aggregation, which lowers the effective contact surface.

Overall, the results highlight the strong dependency of removal efficiency on initial pollutant concentration. While Zn-doped TiO₂ exhibits outstanding photocatalytic activity at low PE loads, its efficiency diminishes under higher concentrations due to surface saturation, light scattering, and ROS competition. This suggests the importance of pre-treatment or dilution strategies for highly contaminated water streams. In natural environments where microplastic concentrations are typically low, such photocatalytic systems offer promising advantages for effective remediation.

Ensuring an appropriate balance between pollutant loading and catalyst dosage is thus essential for cost-effective and high-performance treatment [18][19][20].

3.4 Performance Analysis

A comparative study was carried out to assess the adsorption and photocatalytic efficiencies of TiO_2 , ZnO , and ZnO-TiO_2 catalysts under controlled conditions ($\text{pH} = 3$, catalyst dosage = 3 g/L, PE concentration = 10 mg/L, reaction time = 2 h, temperature = 303 K). The results, presented in Fig. 5, highlight the superior performance of the ZnO-TiO_2 composite across both adsorption and photocatalytic processes. In adsorption studies, ZnO-TiO_2 achieved the highest efficiency (33%), followed by ZnO (30%) and TiO_2 (28%). This trend suggests that Zn doping significantly improves the surface characteristics of TiO_2 , enhancing its affinity toward polyethylene microplastics. The improvement is attributed to the increased surface area, optimized charge distribution, and additional active sites introduced by Zn incorporation. Furthermore, the polar surfaces and amphoteric behavior of ZnO promote stronger interactions with low-density microplastics such as polyethylene under acidic conditions.

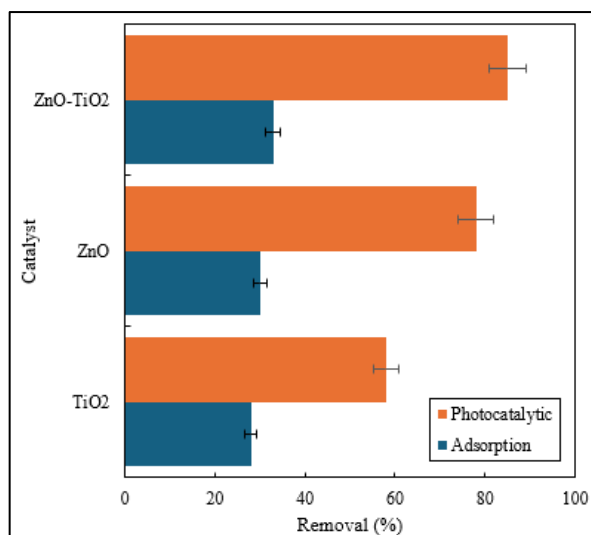


Fig. 5. Performance evaluation of microwave-modified Zn-doped TiO_2 , ZnO , and TiO_2 catalysts [$\text{pH} = 3$, $W = 3$ g/L, $C = 10$ mg/L, $t = 2$ h, $T = 303$ K]

For photocatalytic degradation, the performance gap was more evident. Pure TiO_2 yielded a degradation efficiency of 58%, while ZnO reached 78%, benefiting from its relatively narrower bandgap and superior light absorption in the visible spectrum. The ZnO-TiO_2 composite outperformed both, achieving 85% degradation efficiency. This enhanced activity stems from the synergistic interaction between TiO_2 and ZnO , where Zn doping facilitates effective charge separation and minimizes electron-hole recombination. Additionally, the microwave-assisted synthesis process likely improved crystallinity, particle dispersion, and surface reactivity, collectively enhancing the generation of reactive oxygen species (ROS) under visible light irradiation.

Overall, the combined adsorption and photocatalytic findings demonstrate that ZnO-TiO_2 not only ensures stronger initial binding of microplastics but also promotes more efficient photocatalytic breakdown. These results establish ZnO-TiO_2 as a promising candidate for microplastic remediation, with dual benefits of adsorptive pre-treatment and advanced photocatalytic degradation.

3.5 Reusability Study

The long-term applicability of a photocatalyst in environmental cleanup is governed not only by its activity but also by its stability and recyclability across multiple uses. To evaluate the durability of the microwave-modified Zn-doped TiO₂ catalyst, a reusability test was conducted under controlled conditions (pH = 3, catalyst dosage = 3 g/L, PE concentration = 10 mg/L, reaction time = 2 h, temperature = 303 K). The degradation efficiency was monitored over five successive cycles, as presented in Fig. 6. In the initial run, the catalyst achieved an 85% degradation rate, which was taken as the reference. A slight reduction was noted in the first reuse cycle (83%), with further declines in subsequent cycles: 80% (2nd), 77% (3rd), 75% (4th), and 70% (5th). Such progressive decreases are frequently observed in photocatalyst reuse studies and can be linked to several causes.

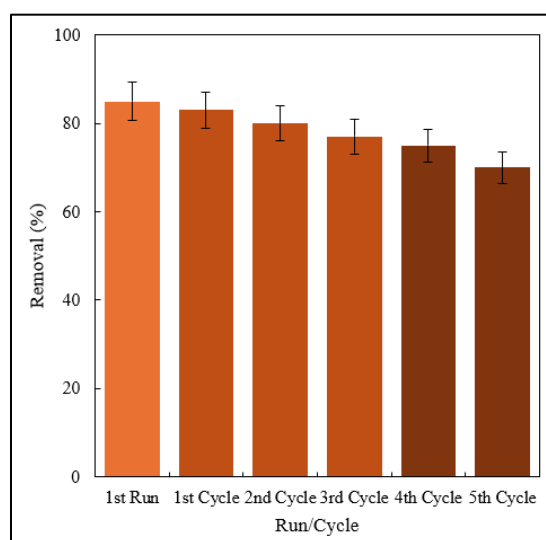


Fig. 6. Reusability performance of microwave-modified Zn-doped TiO₂ catalyst for polyethylene degradation across five cycles [pH = 3, W = 3 g/L, C = 10 mg/L, t = 2 h, T = 303 K].

Performance deterioration is often attributed to surface fouling, where residual polymer fragments or degradation intermediates deposit on the catalyst surface, obstructing active sites and diminishing light penetration. In addition, unavoidable catalyst loss during recovery processes (e.g., centrifugation or filtration) can reduce the effective dosage in subsequent cycles. Possible structural changes, Zn leaching, or surface deactivation caused by photocorrosion may also lower catalytic activity over time [21,22]. Despite this gradual decline, the Zn-doped TiO₂ catalyst maintained more than 82% of its original activity after three cycles and retained 70% efficiency after five cycles. These results highlight the good reusability and stability of the synthesized catalyst, reinforcing its promise for deployment in continuous or semi-continuous water treatment systems targeting microplastic removal.

4. Conclusions

This study demonstrated the effectiveness of microwave-modified Zn-Doped TiO₂ catalysts for the removal of polyethylene (PE) microplastics via combined adsorption and photocatalytic degradation. Catalyst performance was systematically evaluated under varying operational conditions, including pH, catalyst dosage, and initial microplastic concentration. The findings revealed that acidic conditions (pH 3) provided the most favorable environment, yielding the

maximum degradation efficiency of 85%. An optimal catalyst dosage of 3 g/L was identified, as higher loadings led to light scattering and particle agglomeration, thereby reducing activity. Similarly, increasing the initial PE concentration caused a decline in degradation efficiency due to surface saturation, restricted light penetration, and enhanced radical scavenging at higher pollutant loads. Among the catalysts tested, Zn-Doped TiO₂ exhibited superior performance compared to pristine TiO₂ and ZnO, achieving the highest adsorption efficiency (33%) and photocatalytic degradation efficiency (85%). This improvement is attributed to the synergistic effects of Zn incorporation, which promoted visible light utilization and inhibited electron–hole recombination. The reusability study further confirmed the catalyst's robustness, retaining 70% of its degradation efficiency after five consecutive cycles. This stability underscores its practical suitability for continuous or repeated applications. In summary, microwave-assisted Zn-Doped TiO₂ emerges as a promising and sustainable photocatalyst for microplastic remediation, particularly under visible light. These results provide a foundation for future research toward scaling up photocatalytic systems to address persistent plastic pollution in aquatic environments.

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