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Zinc Oxide and Tungsten Trioxide Photocatalysts for Acetaminophen Removal in Water

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Abstract

Untreated Acetaminophen in water is chemically stable and hard to biodegrade. Ozone, ultrasounds, photoelectrochemical oxidation, active carbon adsorption, membrane filtration, and microorganism biodegradation are used to remove Acetaminophen from water. All these methods are effective, but they have drawbacks like incomplete Acetaminophen waste removal, harmful metabolic intermediates, increased water pollution, and the need for additional treatment to remove secondary wastes. Photocatalysis, utilizing light and semiconductors, efficiently removes most wastewater contaminants by decomposing organic compounds. In this study, ZnO, tungsten trioxide (WO_3) and combinations of both were investigated as photocatalysts to degrade Acetaminophen in water. The degradation efficiency was investigated using a glass photoreactor with a working volume of 100 mL and assisted by UV light. Operating parameters, including initial Acetaminophen concentration and photocatalyst dosage, were also studied. The degradation of Acetaminophen was measured using a UV-Vis spectrophotometer. ZnO (80%) shows superior photocatalytic activity for Acetaminophen degradation in water compared to WO_3 (3.45%) and their composites (Z1W1, Z1W3, Z3W1). Adding ZnO into WO_3 enhances its photocatalytic activity and Acetaminophen degradation efficiency compared to only WO_3 as a photocatalyst. The optimal degradation of Acetaminophen in water was achieved at an initial Acetaminophen concentration of 10 mg/L and photocatalyst dosage of ZnO at 0.1g with a degradation efficiency of 92%. Therefore, this proved that ZnO exhibits superior photocatalytic activity in degrading Acetaminophen in water.

1. Introduction

Water pollution emerged as the world's biggest health threat, with a rising prevalence of pollutants such as heavy metals, solvents, sludge and others due to industrialization and population growth [1]. In addition, pharmaceutical residues became a significant concern due to the rise in pharmaceuticals and personal care

products (PPCPs). These pollutants had been discovered in the wastewater streams of hospitals as well as medical factories, which were undeniable contributors to these pollutants. These residues enter the environment via human waste and resist standard wastewater treatment methods [2].

One of the commonly used drugs found in municipal sewage effluent is Acetaminophen [3]. It was ranked among the top 200 most prescribed drugs in the entire world [4]. Acetaminophen is commonly used as a pain reliever, which risks public health as it persists in water sources. Up to 10 mg/L of Acetaminophen has been found in wastewater effluents and 10 ng/L to 100 ng/L in surface water. It had also been found in drinking water at 0.5 ng/L to 45 ng/L as well as in freshwater reservoirs (4.1 ng/L to 73 ng/L) [2]. Its complex structure and stability make it difficult to biodegrade, thereby making it harmful to living organisms. Different methods were explored for removing Acetaminophen from wastewater, such as ozonation, ultrasounds, adsorption, photoelectrochemical oxidation and biodegradation by microorganisms [5]. However, these methods have limitations, including incomplete removal, the generation of harmful intermediates, and increased pollution, necessitating further treatment [6], [7].

Photocatalysis has emerged as a promising method for wastewater treatment, effectively decomposing organic and inorganic compounds with lower energy consumption, such as solar energy, making it an environmentally friendly and sustainable treatment method [5]. The process involves the interaction of a light source with a semiconducting surface, initiating simultaneous oxidation (photogenerated holes, h+) and reduction (photogenerated electrons, e-) processes, leading to the degradation of pollutants [8]. Certain metal oxides in semiconductor photocatalysis, including ZnO, TiO_2, and Fe_2O_3, were used as photocatalysts for organic and inorganic pollutant degradation [9]. Furthermore, Metal-Organic Frameworks (MOFs), graphene-based and bismuth-based photocatalysts have emerged as a new class of photocatalytic materials with unique properties, such as large surface areas, tuneable pore structures, and the ability to incorporate multiple active sites, thus resulting in enhanced light-harvesting capabilities, improving their stability in an aqueous environment and better photocatalytic activity.

ZnO and TiO_2 are widely used as photocatalysts due to their photosensitivity, large band gaps, high efficiency (fast oxidation and reduction rates), and reusability [10]. ZnO, an n-type semiconductor with a band gap of 3.2 eV, showed a promising result in the photocatalytic degradation of volatile organic pollutants [11]. The primary limitation of ZnO in photocatalysis is its restriction to UV light absorption, which limits its efficiency under sunlight. Furthermore, ZnO is prone to photo-corrosion under prolonged irradiation, in which it dissolves in aqueous environments, leading to a loss of material and activity. Therefore, recent research has focused on addressing these challenges through optimization techniques developed for ZnO photocatalysis, including altering its physical properties and semiconductor coupling with other materials (hybrid materials) such as WO_3, thus, enhancing its stability and extending its light absorption to the visible region [9], [11]. To make up for the inadequacies of the individual semiconductors, two (or more) semiconductor photocatalysts are preferable photocatalysts due to their complimentary qualities, relative placements of energy bands, and variations in Fermi level (E_F). This technique encourages the separation of e- and h+ pairs, increases the absorbable spectrum range, and enhances redox ability.

In semiconductor photocatalysis, nano-structural materials were utilized, and photocatalysts like Fe_2O_3/TiO_2, ZnO/TiO_2, $Cu_2O/WO_3/TiO_2$ and $WO_3/TiO_2/SiO_2$ were employed for acetaminophen degradation [12]. ZnO/WO_3 composite photocatalysts demonstrated degradation capabilities for various pollutants such as diclofenac, oxytetracycline, acid red, methyl orange, and Rhodamine B [13], [14]. Combining ZnO and WO_3 in a composite material and heterostructure can create a synergistic effect that enhances photocatalytic performance and leverages the strengths of both materials. The difference in band positions between ZnO and WO_3 allows for effective charge separation, where electrons in the conduction band of ZnO can transfer to the conduction band of WO_3, reducing recombination. This charge transfer mechanism enhances the generation of reactive oxygen species (ROS) and improves the degradation efficiency of pollutants. Additionally, the composite material can utilize both UV and visible light, broadening the range of the solar spectrum that can be harnessed for photocatalysis. For example, ZnO can enhance UV absorption capability, while WO_3 can extend the response to visible light, providing a broader spectrum of activity [15]. Several studies have demonstrated the enhanced photocatalytic performance of ZnO/WO_3 composites in the degradation of pharmaceuticals [16], [17]. For instance, research on the degradation of diclofenac, tetracycline and other common pharmaceutical micro-pollutants has shown that ZnO/WO_3 composites achieve higher degradation rates compared to individual ZnO or WO_3. However, there are few studies on the photocatalysis of these materials on other micropollutants such as Acetaminophen. Considering these findings, the degradation efficiency of Acetaminophen using ZnO and WO_3 was investigated in this study using UV-based photodegradation. The influence of different physicochemical parameters, such as ZnO/WO_3 molar ratios, initial Acetaminophen concentration and catalyst loading, on the Acetaminophen degradation performance was also explored. Thus, the potential of ZnO/WO_3 composites could offer a highly effective approach for the photocatalytic degradation of pharmaceutical pollutants in wastewater, which further enhances their performance and practical applicability.

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2. Experimental

2.1 Preparation of Acetaminophen Solutions

A stock solution of Acetaminophen with a concentration of 500 mg/L was prepared by dissolving one Panadol Soluble tablet (500 mg) that had been crushed into powder into 1 L of distilled. The mixture was stirred continuously until all the Panadol powder dissolved. The stock solution of Acetaminophen was then diluted (working solutions: 5 mg/L, 10 mg/L, 15 mg/L, 20 mg/L, and 25 mg/L) when required for the experiments. Both stock and working solutions were placed in a box (dark place) to prevent the degradation of the solutions by visible light. This precaution ensured the stability and integrity of the solutions, maintaining their desired properties and effectiveness for usage in the following experiments.

2.2 Preparation of WO_3 and ZnO/WO_3 Photocatalysts

WO_3 photocatalyst was prepared by adding 2.5 g of H_2WO_4 (Sigma-Aldrich, purity ~99%) powder into 100 mL of H_2O_2 (R&M Chemical, purity 30-35%) in a beaker and stirring it with a magnetic stirrer at room temperature until the H_2WO_4 powder completely dissolved. After the H_2WO_4 powder dissolved, the solution was transferred into an oven and left overnight at 60 °C to promote the synthesis process. Subsequently, the solution was transferred into a furnace and heated for one hour at 500 °C to further enhance the formation of the WO_3 photocatalyst. Finally, the resulting material was ground and carefully transferred into a test tube for further application. ZnO/WO_3 photocatalysts with different molar ratios specified in Table 1 were synthesized by mixing ZnO (Sigma-Aldrich, purity ~99%) and H_2WO_4 powder into 100 mL H_2O_2. Then, the steps of synthesization followed the standard procedures similar to the preparation of WO_3 photocatalysts. These photocatalysts were used to assess the physicochemical influence on the photodegradation of Acetaminophen, as explained in the following section. The prepared photocatalysts were stored in a desiccator to maintain the condition and performance of the catalyst by preventing exposure to air or moisture or undergoing undesired reactions.

Table 1 *Variation of molar ratio for ZnO/WO_3 photocatalyst synthesized for this study*

ZnO (g)	WO_3 (g)	Photocatalyst Molar Ratio
1	1	1:1
0.5	1.5	1:3
1.5	0.5	3:1

2.3 Photodegradation Experiments

To evaluate the photocatalytic activity of Acetaminophen in an aqueous solution, photocatalysis experiments were carried out in a glass photoreactor with a capacity of 250 mL and assisted by UV light (see Fig. 1). In the photoreactor, a predetermined quantity of the photocatalyst powder was added to 100 mL of Acetaminophen (working volume) with a specified concentration. Before the photodegradation experiment, the photoreactor was kept in the dark for 30 minutes to allow the adsorption-desorption equilibrium to reach a steady state. Air was supplied continuously to the photoreactor at a constant flow rate of 4 L/min to maintain dissolved oxygen in the solution. Simultaneously, the mixture was stirred using a magnetic stirrer and illuminated by UV light for 150 minutes. After the photodegradation process, the photocatalyst was filtered using filter paper. The filtrate was collected and analyzed using a UV-VIS spectrophotometer. A calibration curve (absorbance vs Acetaminophen concentration) was prepared prior to measuring Acetaminophen concentration in the experiments. The percentage of Acetaminophen degradation in water was calculated using Eq. (1).

$$\text{Percentage of Degradation (\%)} = \frac{C_o - C_t}{C_o} \times 100 \quad (1)$$

where C_o represents the concentration of Acetaminophen before irradiation, and C_t represents the concentration of a pollutant at t minutes.

2.4 Physicochemical Influence on Photocatalytic Activity

The physicochemical parameters that affect the photocatalytic activity of the photocatalysts for Acetaminophen degradation investigated in this study were molar ratio, dosage, and initial Acetaminophen concentration. The photocatalysts used in this study were the individual and combinations of ZnO and WO_3 by molar ratio (w/w) as follows: ZnO/WO_3 (Z1W1), $3ZnO/WO_3$ (Z3W1) and $ZnO/3WO_3$ (Z1W3); to determine the best photocatalyst

ratio based on their photocatalytic activity. ZnO and WO_3 were also investigated as indicators for their individual effect on the photodegradation of Acetaminophen. The effects of the initial concentration of Acetaminophen (5, 10, 15, 20, and 25 mg/L) and photocatalyst dosage (0.1, 0.2, 0.3, 0.4, and 0.5 g) on photocatalytic activity were also carried out. The parameters were maintained at 10 mg/L (initial concentration of Acetaminophen) and 0.2 g (dosage), otherwise required.

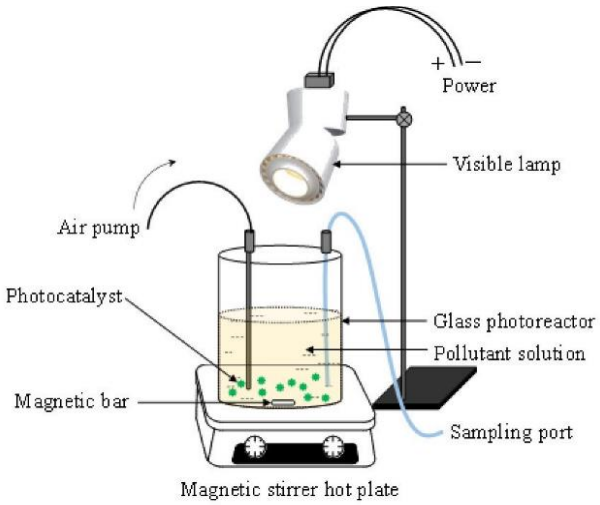

Fig. 1 *Schematic illustrations of the photoreactor setup for degradation of Acetaminophen in aqueous solution using ZnO/WO_3 photocatalyst*

3. Results and Discussion

3.1 Calibration Curve

A calibration curve of absorbance (AU) vs Acetaminophen concentrations (5 to 25 mg/L) was plotted (see Fig. 2). A linear curve is obtained with R^2 = 0.9996. This curve was used to measure the concentration of Acetaminophen after its photodegradation process in the following experiments.

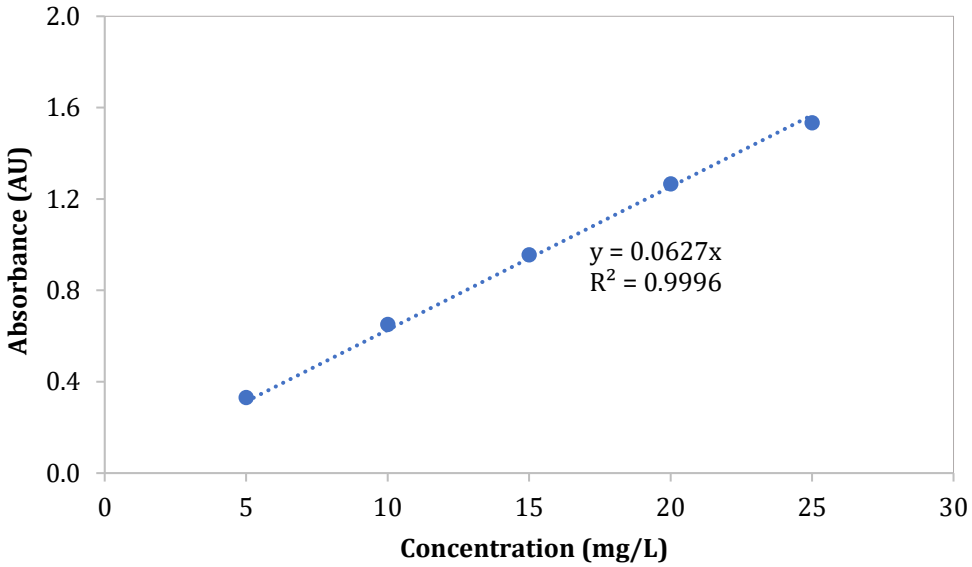

Fig. 2 *Calibration plot of acetaminophen*

3.2 Types of Photocatalysts for Acetaminophen Degradation

Fig. 3 shows the performance and synergistic effects of the ZnO/WO_3 photocatalyst, as well as the inherent capabilities of each component when used independently. The results show that ZnO (80%) has a superior photocatalytic activity for Acetaminophen degradation in water compared to WO_3 (3.4%). This may be due to

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the larger band gap (~3.37 eV) of ZnO compared to WO3 (~2.6 eV), which allows it to absorb more energy from UV light and generate more electron-hole pairs for the photocatalytic process, thereby degrading pollutants more efficiently under UV light [18]. Furthermore, ZnO has higher electron mobility, which improves charge-carrier separation and reduces recombination rates, thereby increasing the concentrations of electrons and holes for redox reactions [19], [20]. Although WO_3 also exhibits photocatalytic activity, its narrower bandgap (~2.6–2.8 eV) makes it more responsive to visible light. However, this also means that the photogenerated electrons in WO_3 are less energetic than those in ZnO, potentially making WO_3 less effective in driving certain oxidative degradation processes, such as the breakdown of Acetaminophen [21].

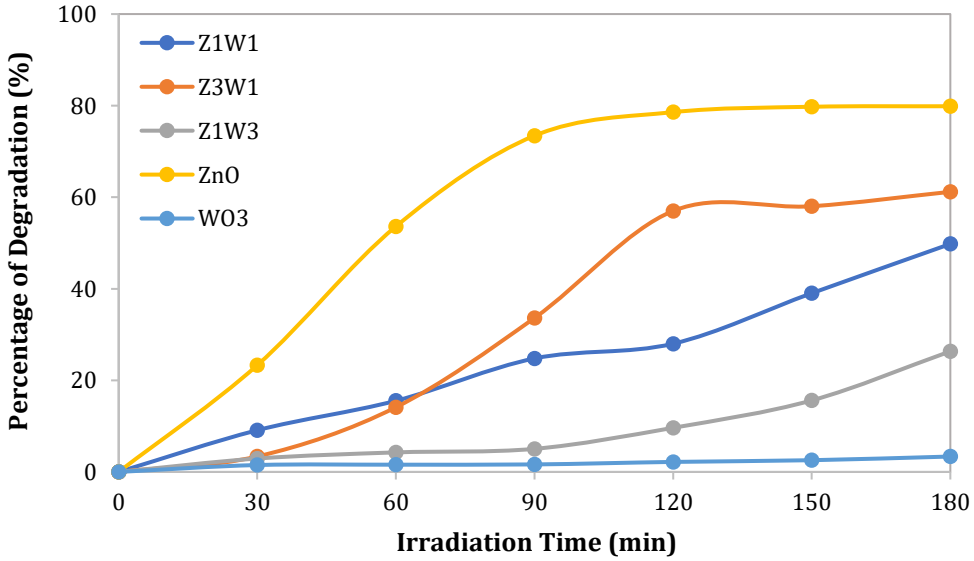

Fig. 3 *Graph for percentage of degradation versus irradiation time for five different photocatalysts*

The combination of a higher ZnO ratio with WO_3 (Z3W1) also achieved the highest Acetaminophen degradation of 61 %. The ZnO/WO_3 (1:3) composition had a lower degradation percentage of 26.34%, suggesting an excess of WO_3 reduces the composite efficiency for Acetaminophen. This agrees well with the degradation performance of methylene blue and anionic orange reported by Adhikari et al. [15], which achieved <50% degradation efficiency when the WO_3 ratio exceeded that of ZnO. These findings indicate that the photocatalytic performance of ZnO/WO_3 composite is highly dependent on the ZnO amount, which affects their photocatalytic activity. The selection between ZnO and WO_3 is highly contingent on specific applications and operational conditions. WO_3 possesses several advantages that make it suitable for particular use, notably its better absorption in the visible light range (400-700 nm). This characteristic can significantly enhance the efficiency of visible-light-driven photocatalysis, which is particularly beneficial in applications where the utilization of solar energy is critical [22]. Since ZnO demonstrates superior photocatalytic activity for Acetaminophen degradation compared to WO_3 and their combinations, ZnO was selected as a photocatalyst in the following experiments.

3.3 Effect of Initial Concentration for Acetaminophen Photodegradation

The variation in Acetaminophen concentrations (5 to 25 mg/L) was used to determine the correlation of photocatalytic activity of ZnO reaction on different amounts of Acetaminophen in the solution. Fig. 4 shows a gradual increase in Acetaminophen degradation, followed by a constant value upon reaching equilibrium after 90 to 120 minutes of reaction time, for all Acetaminophen concentrations. The gradual increase in pollutant photodegradation at the beginning of a photocatalytic reaction can be attributed to numerous active sites on the photocatalyst that are available for pollutant adsorption. The higher concentration of pollutants at the start enhances the probability of collisions with these active sites, leading to increased degradation rates [23]. Additionally, the generation of reactive species (such as hydroxyl radicals) and efficient light absorption by the photocatalyst at the onset contribute to the rapid initial photodegradation [7]. The maximum Acetaminophen degradation (~80%) was achieved at 10 mg/L within 90 minutes of reaction time. The initial concentration of 15 mg/L showed a similar photodegradation efficiency of 79%. Lower initial concentrations are more favourable for efficient photodegradation with ZnO as the photocatalyst, potentially due to competition for reactive sites or increased light absorption.

3.4 Effect of Photocatalyst Dosage for Acetaminophen Photodegradation

The effect of ZnO loading on the photocatalytic degradation of Acetaminophen was determined by systematically varying the photocatalyst loading between 0.1 and 0.5 g. The optimal photocatalyst loading for Acetaminophen degradation in water was found to be 0.1 g with a degradation efficiency of 92.06% (see Fig. 5). Higher loadings of 0.2 – 0.5 g, 0 resulted in lower degradation percentages (80 - 89%), indicating a decrease in efficiency. This may be due to light scattering, reduced exposure to UV light, self-shading, particle aggregation and mass transfer limitation, which limit photocatalytic activity. Based on these considerations, loading of 0.1 g provides a better balance between photocatalyst amount and degradation efficiency, while minimizing the negative effects associated with higher loadings [24].

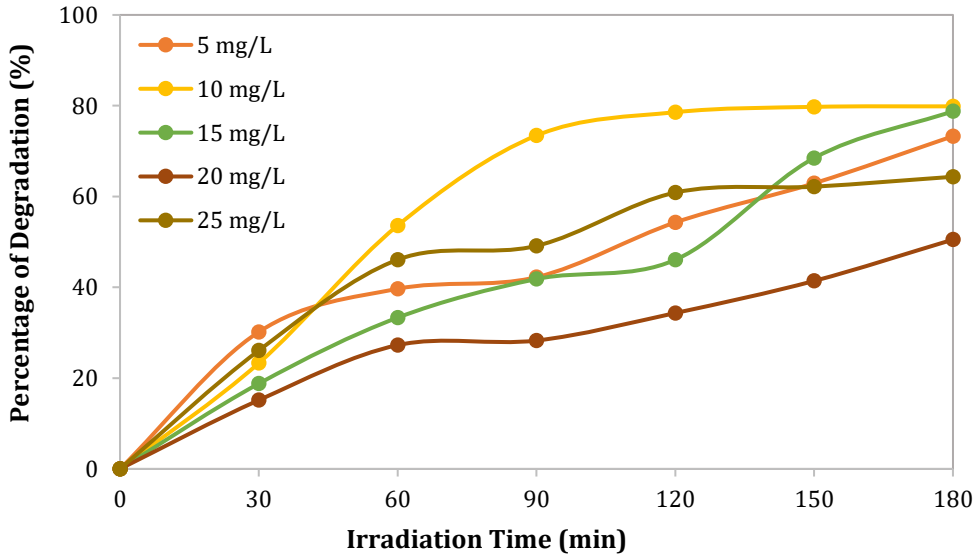

Fig. 4 *Effect of initial concentration on photodegradation of Acetaminophen*

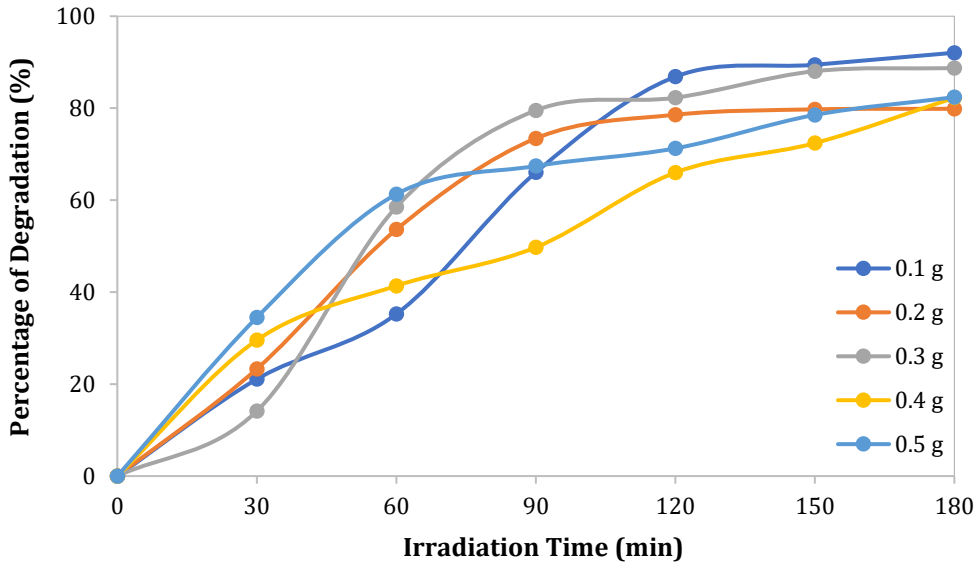

Fig. 5 *Effect of photocatalyst dosage on photodegradation of Acetaminophen*

3.5 Optimizing Acetaminophen Photodegradation

The optimal photodegradation of Acetaminophen was assessed using conditions with maximum degradation efficiency established from previous experiments, at a photocatalyst dosage of 0.1 g using ZnO as the photocatalyst. Two initial Acetaminophen concentrations of 10 and 15 mg/L were compared since both gave almost similar photodegradation efficiency (~80%). Fig. 6 shows photodegradation efficiency of Acetaminophen was higher at 10 mg/L (92%) compared to 15 mg/L (86.4%). However, at both concentrations,

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ZnO could degrade more than 85% of Acetaminophen using considerably lower dosage. This shows a promising result and is comparable to the Acetaminophen photodegrading efficiencies obtained by different photocatalysts as shown in Table 2.

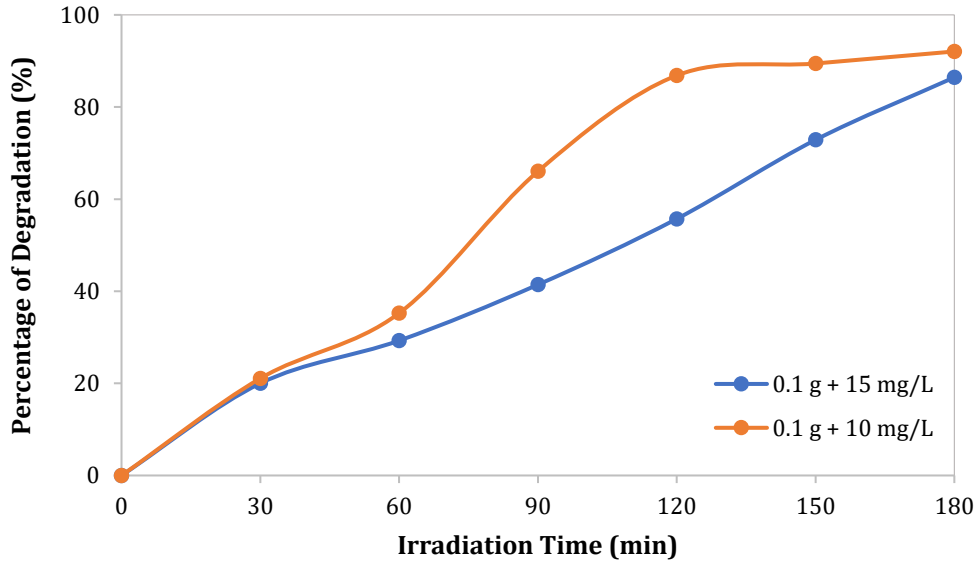

Fig. 6 *Graph for percentage of degradation versus irradiation time for combined optimum parameters for 0.1 g of ZnO in 10 and 15 mg/L of Acetaminophen*

Table 2 *Acetaminophen photodegradation efficiency using different types of photocatalysts*

Type of Photocatalyst	Initial Concentration of Acetaminophen (mg/L)	Dosage of Photocatalyst (g/L)	Source of Light (intensity)	Irradiation time (minutes)	Degradation efficiency (%)	References
ZnO	20	2	Solar simulator	60	100	[7]
TiO_2	60	1	Natural solar light	210	88	[26]
$BaTiO_3/TiO_2$	5	1	Xenon lamp (500 W)	240	95	[25]
Fe_2O_3/TiO2	30	1.25	Solar simulator	180	95.8	[4]
$WO_3/TiO_2/SiO_2$	5	1.5	Xenon lamp (500 W)	240	95	[3]
ZnO	10	1	UV light	120	92	This study

4. Conclusion

The photocatalysis experiments carried out in this study show ZnO was the superior photocatalytic activity compared to WO_3 and their composites, for the degradation of Acetaminophen in water. The addition of ZnO to WO_3 enhanced photocatalytic activity, leading to higher degradation percentages. The initial Acetaminophen concentration of 10 mg/L and 0.1 g of ZnO achieved the optimal Acetaminophen degradation at 92%. Higher concentrations of Acetaminophen hindered the photocatalytic process due to the consumption of reactive species and saturating the active sites on the photocatalyst surface, while lower concentrations of Acetaminophen limit the availability of Acetaminophen molecules for efficient degradation. Moreover,

photocatalyst loading of 0.1 g was found to be the most advantageous, allowing better utilization of the photocatalyst and its surface area, leading to more efficient Acetaminophen degradation.

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Conflict of Interest

There is no conflict of interest regarding the publication of the paper.

Author Contribution

The authors confirm their contribution to the paper as follows: ***Study conception and design:*** *Muhammad Izuan Atiq Zainudin, Hamizah Mokhtar, Zadariana Jamil;* ***Data collection:*** *Muhammad Izuan Atiq Zainudin;* ***Analysis and interpretation of results:*** *Muhammad Izuan Atiq Zainudin, Hamizah Mokhtar, Zadariana Jamil;* ***Draft manuscript preparation:*** *Muhammad Izuan Atiq Zainudin, Zadariana Jamil, Azianabiha A Halip Khalid, Nur Kamaliah Mustaffa. All authors reviewed the results and approved the final version of the manuscript.*

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