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Photodegradation of Bisphenol A using WO_3/ZnO Photocatalyst under Visible Light Irradiation

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ABSTRACT. The issues on the safety and dwindling of water quality due to the presence of exogenous endocrine-disrupting chemicals (EDCs) such as Bisphenol A (BPA) have become a major toxicological threat and growing concern. In a bid to curb the pollutants accumulation in water, semiconductor-mediated photocatalysis has been shown to be better suited for the removal of such organic pollutants because the technique offers a green and sustainable process by utilizing natural solar light as the energy source to mitigate the pollutants. ZnO has formerly been used as a photocatalyst, but this material has a wide band gap of 3.37 eV that can only be fully employed under ultraviolet irradiation and accounts for less than 5% of solar radiation. Therefore, the doping of low band gap material such as WO_3 into ZnO lattice could provide ideal surface characteristics enhancement for BPA degradation. The coupling could maximize the redox potential in which the flow of electrons from the conduction band of WO_3 to ZnO is expected. In this study, WO_3/ZnO composite photocatalyst was synthesized at various mol ratios via simple chemical precipitation method. The degradation of BPA in aqueous solution under visible light irradiation (23 W) over 0.5 g of WO_3/ZnO (1:3) composite photocatalyst was shown to achieve the best degradation percentage of 65.73% within 180 min.

Keywords: Bisphenol A, Photocatalyst, Tungsten trioxide, Visible light, Zinc oxide

INTRODUCTION

The recent energy crisis and environmental pollution had a significant negative impact on both human health and environmental ability in order to develop the sustainability of the environment. These issues are directly related to the world's rapid population growth and the drastic industrial development (Wang et al., 2022). Water pollution by a variety of exogenous endocrine-disrupting chemicals (EDCs) from various industries has become an eco-toxicological hazard of prime interest and increasing significance. The ubiquitous presence of such chemicals in water bodies, even at a very low concentration (ngL^{-1}), has seriously threatened the survival of aquatic organisms and humans (Aris et al., 2020). Particularly, Bisphenol A (BPA) is a product of synthetic compounds that have been widely used nowadays, especially in the production or manufacture of plastics such as polycarbonate plastics and resins epoxy. Unfortunately, BPA is considered to have negative effects on health and has been considered an environmental

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pollutant (Reddy et al., 2018).

The removal of BPA is challenging and complicated due its unique chemical and physical characteristics and its ability to interfere with hormonal systems. Photocatalysis became an effective and low-impact technique for the removal of pollutant organics and non-biodegradable compounds from the aqueous solution of wastewater (Madkhali et al., 2023). In this perspective, semiconductor-mediated photocatalysis has been proposed as an effective technique to combat the toxic and hazardous organic pollutants in EDCs-containing wastewater. This system has been a promising technique since it is inexpensive and safe, which requires the use of efficient light-harvesting, environmentally benign, and non-toxic semiconductor materials such as zinc oxide (ZnO), a semiconductor as the photocatalyst to degrade the pollutants (Kubacka et al., 2021). Nonetheless, a major downside of pristine ZnO is its large band gap (3.37 eV), which means it can only be fully exploited under ultraviolet (UV) region ($\lambda \leq 400$ nm), thus limiting the practical efficacy for visible/solar applications (Shohany and Zak, 2020). In this view, it is imperative to construct a photocatalyst so that it can be fully exploited under visible or solar light. Improving photocatalytic activity and optimization of ZnO is possible by modification of its surface and electronic properties through doping with different materials. Currently, increasing attention has been paid to the doping of ZnO with other semiconductor oxides (heterojunctions) since it provides a promising way to avoid deteriorating thermal stability of the ZnO lattice crystalline structure, improving Fermi level alignment, restricting electron-hole recombination, narrowing the band gap, and shifting the absorbance to the visible region ($\lambda \geq 400$ nm) (Low et al., 2017).

In a previous study, the doping of TiO_2 with ZnO lattice crystals led to a narrowed band gap and facilitated visible light absorption capability of the methylene blue (MB) and methylene red (MR) dyes degradation (Mousa et al., 2021). Meanwhile, Mano et al. attributed the aniline and formaldehyde degradation enhancement of ZnO-PbS to the ZnO acting as an electron extractor from the PbS conduction band, resulting in the increase of the charge carriers' lifetime (Mano et al., 2020). In this context, the abundant transition metal compounds, especially tungsten oxide (WO_3), are materials of interest with promising potential for many technological applications. WO_3 is an n-type semiconductor that presents unique optical and electronic properties (band gap of 2.8 eV) that can be explored for the development of heterojunctions and a series of functional structural phases (Liu et al., 2021). In order to improve the performance of ZnO, the coupling of low band gap materials such as WO_3 with ZnO to form heterostructures is one of the most important approaches to band gap engineering, which can enhance photocatalytic properties at the visible region. Thus, the emergence of the heterojunction's mechanism could solve the problem and maximize the redox potential to induce the flow of electrons from the conduction band of WO_3 to ZnO and vice-versa. As a result, the photogenerated electrons and holes can be spatially distributed in two different crystalline phases of ZnO and WO_3, and charge recombination is inhibited, subsequently contributing to the efficient photocatalytic activity towards organic pollutants in a timely manner in comparison to the performance of the isolated oxides.

In this paper, we present a straightforward fabrication procedure for WO_3/ZnO photocatalysts by changing the mol ratios of both materials using a simple chemical precipitation approach. Emphasis was placed on the effect of the formulation, physicochemical properties, and synergistic interaction between WO_3 and ZnO to facilitate the charge carrier transfer. The morphological and surface compositions of the WO_3/ZnO photocatalysts were examined using

scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analyzer. The photocatalytic performances of the prepared composites were tested against Bisphenol A (BPA) in an aqueous solution assisted by a very low intensity of visible light irradiation (23 W) for a period of 180 min. Furthermore, the effects of several parameters, such as catalyst dosage and initial BPA concentration, were also assessed to evaluate the feasibility of the synthesized photocatalyst in different environments. However, to the best of our knowledge, there is yet any research that attempts to prepare WO_3/ZnO photocatalyst for BPA removal under low visible light intensity, and it is expected that incorporating the suitable amount of WO_3 and ZnO could produce an optimum photocatalytic performance for practical water remediation process.

METHODOLOGY

Materials

Tungstic acid (H_2WO_4, ≥ 99%), zinc oxide (ZnO, ≥ 99%), and Bisphenol A (BPA, 97%) were purchased from Sigma Aldrich (Selangor, Malaysia). Hydrogen peroxide (H_2O_2, 30-35%) was obtained from R&M Chemicals (Selangor, Malaysia). All chemicals were applied as received, and deionized water was used throughout the experimental procedures.

Preparation of WO_3/ZnO composite photocatalysts

The preparation of WO_3/ZnO photocatalysts was carried out via a facile chemical precipitation approach. Firstly, 2.5 g of H_2WO_4 was dissolved in 100 mL H_2O_2 to form a colourless and stable peroxo-polytungstic acid solution at room temperature. Subsequently, 0.8 g of zinc oxide (ZnO) nanoparticle was added as a pre-formed particle with continuous stirring for 1 h to obtain heterostructures utilizing the stable peroxo-polytungstic acid solution. The solution mixture was then sonicated for 1 h before drying in the oven at 80°C overnight. The obtained WO_3/ZnO composite (WO_3 to ZnO mol ratio = 1:1, denoted as W: Zn = 1:1) was calcined at 500°C for 1 h with a heating rate of 5°C/min (Gao et al., 2017). The obtained powder was characterized and used for photocatalytic reaction. The WO_3/ZnO composite with other mol ratios of WO_3 to ZnO (1:3,1:5, 3:1, and 5:1) was synthesized according to the typical procedure described above. The same procedure was performed without ZnO to obtain only WO_3 particles and for comparison purposes. A summarized denomination of each material is presented in Table 1.

Table 1. The variation of mol ratios for the preparation of WO_3/ZnO heterojunction photocatalyst.

Tungstic acid, H_2WO_4 (mol)	Zinc Oxide, ZnO (mol)	Labeled as
0.01	0.01	WZ (1:1)
0.01	0.03	WZ (1:3)
0.01	0.05	WZ (1:5)
0.03	0.01	WZ (3:1)
0.05	0.01	WZ (5:1)

Evaluation of the photocatalytic performance of WO_3/ZnO composite photocatalysts

Prior to the photocatalytic activity testing, the calibration plot of the targeted pollutant (Bisphenol A, BPA) in an aqueous solution under normal conditions was performed. The calibration curve was plotted according to the various concentrations of the targeted pollutant, ranging from 5 to 25 mg/L. From this calibration plot, the linear equation was determined by measuring the absorbance of each sample at the maximum wavelength (λ_{max}= 275 nm). The photocatalytic performance of BPA in an aqueous solution was conducted in a 250 ml glass photoreactor assisted by a 23 W fluorescence lamp as a visible light source. A 0.2 g of the powder photocatalyst was added to the photoreactor containing a known concentration of the working solution (100 mL). The photoreactor was constantly supplied with air to warrant the presence of dissolved oxygen in the solution with a fixed flow rate of 4 L/min and left in the dark for 30 min before starting the photodegradation testing to achieve adsorption-desorption equilibrium. The photoreactor was then exposed to visible light irradiation. The liquid (5 mL) was collected at a predetermined time interval and filtered to remove the photocatalyst. The effect of mol ratio variation in WO_3/ZnO composite photocatalysts for photodegradation of BPA was monitored by using a Perkin Elmer UV-visible spectrophotometer. In this study, the degradation percentage was calculated using the following equation.

$$Degradation\ percentage\ (\%) = \frac{(C_O - C_t)}{C_O} \times 100\% \quad (1)$$

where C_o is the concentration of BPA before irradiation, and C_t is the concentration of BPA at a time 't'.

Characterizations

The structural and elemental compositions were characterized using a scanning electron microscope equipped with an electron-dispersive X-ray analyzer (SEM-EDX, TESCAN VEGA3).

RESULTS AND DISCUSSION

Characterization

The morphological characteristics of each photocatalyst were observed by using scanning electron microscopy (SEM). Meanwhile, energy dispersive X-ray analysis (EDX) has also been used to allocate the elemental information on the surface of the composite photocatalyst. Figure 1 shows the morphological surface of pure WO_3, pure ZnO, and WO_3/ZnO composite photocatalysts prepared with different mol ratios which were recorded at 5000x magnification. Pure WO_3 exhibited a denser and compact surface (Figure 1a), while pure ZnO displayed a homogeneous quasi-spherical shape (Figure 1b). It is clearly seen that a similar quasi-spherical shape was formed for WO_3/ZnO (1:1) in which the surface of WO_3 was completely covered by ZnO. The particles were observed to be well dispersed on the surface, like pure ZnO (Figure 1c). As the ZnO contents increased up to 0.05 mol, the composite photocatalysts further exposed the protruding surface of WO_3 with a larger particle size than ZnO (Figure 1d and e). Moreover,

increasing the WO_3 contents further produced agglomerated particles, and the presence of ZnO particles was not clearly shown (Figure 1f and g), which is due to the calcination at 500°C for 1 h. Figure 2 shows the elemental composition of the WO_3/ZnO (1:3) composite. The particles, which are represented by the colours green for Zn, red for W, and blue for O in the mapping images, have good dispersion. The data showed that Zn, W, and O were present on the photocatalyst surface, with Zn dominating the composition with an atomic percentage of 48.65%, followed by W (28.92%) and O (22.43%). The absence of other peaks in the EDX pattern and the Au peak's connection to the gold coating used for SEM images serve to verify the samples' purity.

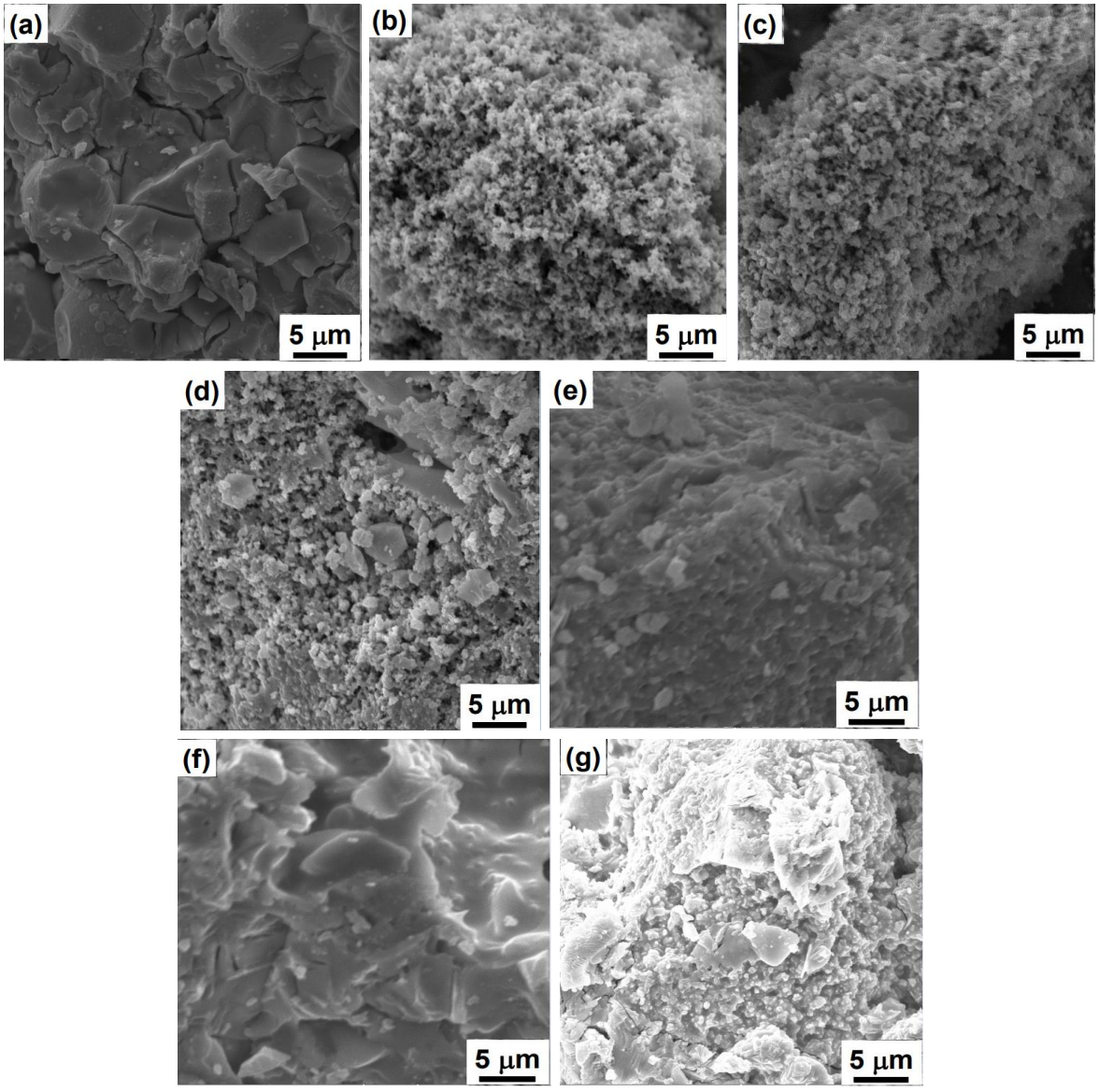

Figure 1. SEM images of (a) pure WO_3, (b) pure ZnO, (c) WO_3/ZnO (1:1), (d) WO_3/ZnO (1:3), (e) WO_3/ZnO (1:5), (f) WO_3/ZnO (3:1), and (g) WO_3/ZnO (5:1) composite photocatalysts at 5kx magnification

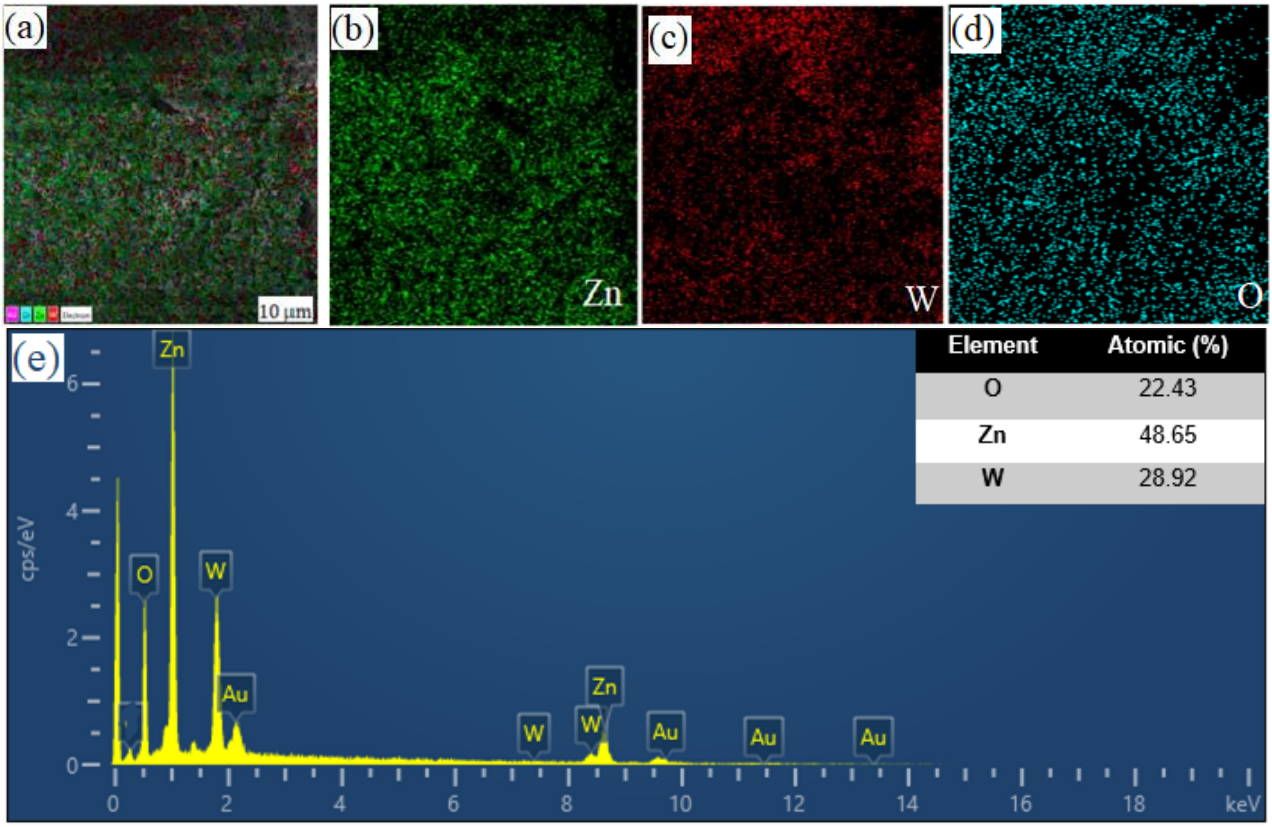

Figure 2. EDX analysis of (a) selected image of the WO_3/ZnO (1:3) composite photocatalyst with corresponding elemental mapping images for (b) Zn, (c) W, (d) O, and (e) the elemental spectra and sample's composition

Photocatalytic performance

The evaluation of the photocatalytic performance of the composite photocatalysts toward Bisphenol A (BPA) was carried out under visible light irradiation for 180 min. In the absence of a photocatalyst (photolysis), 5.7% of BPA was degraded, and this value seems to be negligible, as shown in **Figure 3**. The direct photolysis of BPA suggested that the molecule was considerably stable and would not degrade without the presence of a photocatalyst. On the other hand, WZ (1:3) gave the highest degradation percentage of 30.33%, followed by WZ (1:1), which exhibited a slightly lower degradation percentage (22.10%). However, when the BPA was treated with WZ (1:5) and WZ (3:1), the degradation percentage decreased to 17.18% and 16.2%, respectively, with WZ (5:1) giving the lowest degradation percentage of 14.4%. Photocatalyst band gap plays the most important role, especially in the photocatalytic degradation of organic pollutants that involve BPA. This occurred when the photocatalyst had irritated and gave a response towards irradiation of visible light. The pair of electron-holes would be generated. Thus, those reactive species react and interact with the organic pollutants in order to break down them into much simpler compounds (Madkhali et al., 2023). Unfortunately, if the band gap produced by the photodegradation process is too large, the amount of energy acquired to generate pairs of electron-hole would also be a huge amount, which might be able to lead limited efficiency in the process of photodegradation (Modwi et al., 2018). This clearly shows that with the smaller sizes of band gaps produced by photocatalysts, the energy acquired to generate the highly reactive pairs of electron-hole is much lower. This also might lead to the much higher adsorption of irradiation visible light, which

is able to enhance the efficiency process of photocatalytic degradation (Noureen et al., 2022). It was observed that the photocatalytic performance was influenced by the gap energy of the respective photocatalysts (Liu et al., 2021).

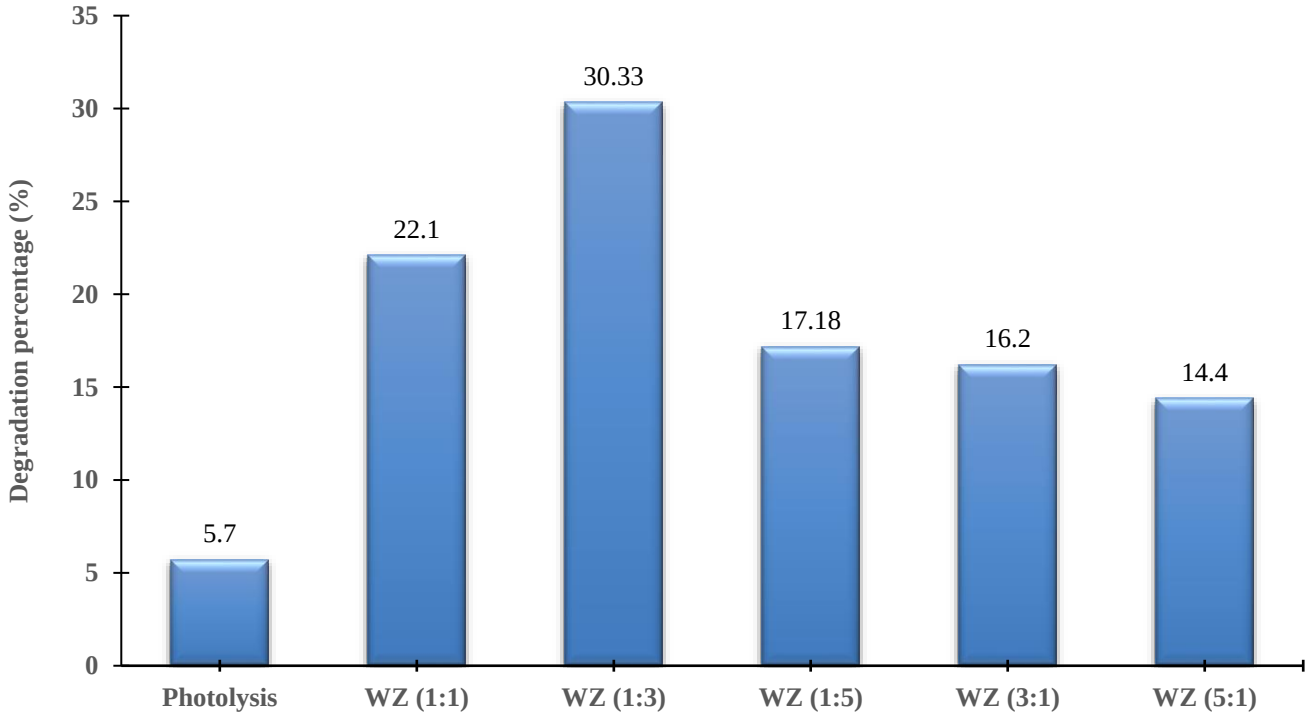

Figure 3. Photolysis and degradation of BPA in aqueous solution by using different mol ratios of WO_3/ZnO composite photocatalysts. Conditions: [BPA] = 10 mg/L, dosage = 0.2 g, irradiation time = 180 min, pH = 2.

Theoretically, when the catalyst particles are exposed to light irradiation (photons) with energy equal to or higher than its band gap, it results in the formation of photoinduced electrons (e^-) and positive holes (h^+) in the conduction and valence bands, respectively. These species were further reacted in a series of redox reactions between adsorbed oxygen and adsorbed water to produce •OH radicals (Mohd Hir and Ali, 2021). The formation of this powerful oxidizing agent (•OH) would have the aptitude to attack the BPA compounds present at the surface of the photocatalyst (Shandilya et al., 2022). Consequently, it mineralized the toxic compounds into harmless species such as H_2O and CO_2 molecules. Furthermore, the results indicate that higher photocatalytic activity exhibited by WZ (1:3) photocatalyst might be due to the increase of the charge separation by extending the energy range of photoexcitation. Subsequently, their characteristics or physical properties are significantly modified. Therefore, such factors might contribute to the increase in the percentage of photodegradation. One of the factors that might affect the rate of degradation is photocatalyst loading influences. The photocatalyst loading is able to provide a higher number of active sites, and this led to enhancing the performance of photodegradation. Most researchers have stated in previous investigations that the performance or role of catalyst loading is able to influence the rate of degradation (Bedia et al., 2019; Samuel et al., 2022). On the other hand, the lower degradation rate obtained by WZ (5:1) is probably due to the fast recombination process occurring between the photogenerated electron-hole pairs in the catalyst itself, which has high band gap energy. On the contrary, increasing the amount of catalyst that is beyond the optimum consumption potentially would drop the performance of the degradation rate. This occurred due to the over amount

of agglomeration, which is also known as the interaction of the particle-site that might generate a high amount of effluent turbid (Razali et al., 2021). This was in good agreement as reported by Ruan et al. (2020) on the degradation of Acid Orange 7 using binary photocatalyst of ZnO/CuO.

The photodegradation of BPA was further studied by varying its initial pH from pH 2 to pH 10 under visible light irradiation. Figure 4 shows that the photocatalyst was significantly affected in acidic conditions as compared to basic conditions under visible light irradiation. In the WZ (1:3) photocatalytic system, the degradation percentage of BPA decreases gradually with increasing pH solution up to pH 10. This occurrence can be attributed to the surface charge of ZnO and WO_3 at different pH. The charged surface of the photocatalyst and the constant dissociation of the acidic concept of wastewater might be influenced by the solution pH (Razali et al., 2021). The electrostatic attraction in acidic conditions results in a higher rate of reaction and better photocatalytic performance. The best degradation percentages were found at pH 2, and when the reaction solution's pH was raised from 2 to 10, the photodegradation results were decreased. Because methylene blue is cationic, at lower pH levels, methylene blue molecules absorb on the catalyst surface, increasing the photocatalytic performance. A similar trend was reported by Akhter et al. (2023), where lowering the solution pH seemed to favour the degradation activity in which the surface of the methylene blue (MB) was attracted to the TiO_2 and ZnO surfaces, hence yielding a higher degradation percentage.

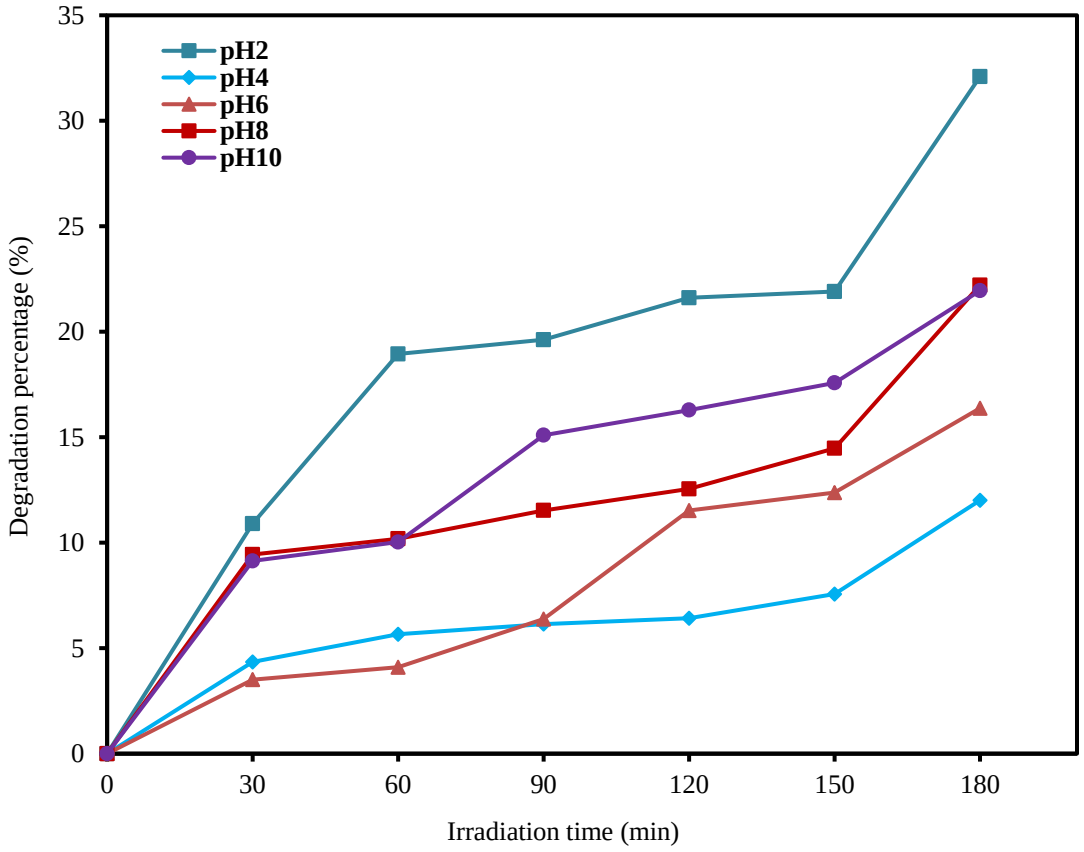

Figure 4. Degradation percentage of BPA with different initial pH under visible light irradiation by using WO_3/ZnO (1:3) composite photocatalyst. Conditions: [BPA] = 10 mg/L, dosage = 0.2 g.

The effect of photocatalyst dosage under visible light irradiation by using WZ (1:3) composite photocatalyst had a major impact on the photocatalysis process. The dosage ranging from 0.1 to 0.5 g was used for the degradation of BPA (Figure 5). The photocatalytic performance of the best composite photocatalyst increased with an increasing amount of dosage up to 0.5 g, yielding the highest degradation percentage of 65.73%. This was attributed to the increase in the active sites for the sufficient absorption of photon energy from the visible light irradiation. It is evident that as catalyst concentration was increased, surface active sites became more available. As a result, the number of reactive species (•OH, $•O_2^-$, h^+, e^-) responsible for BPA's degradation also rose. However, there is also a possibility that at a certain limit, the degradation rate could be slowed down as a result of the particles' tendency to aggregate, which reduces the number of surface-active sites and will lead to ineffective light absorption (Zhu et al., 2017).

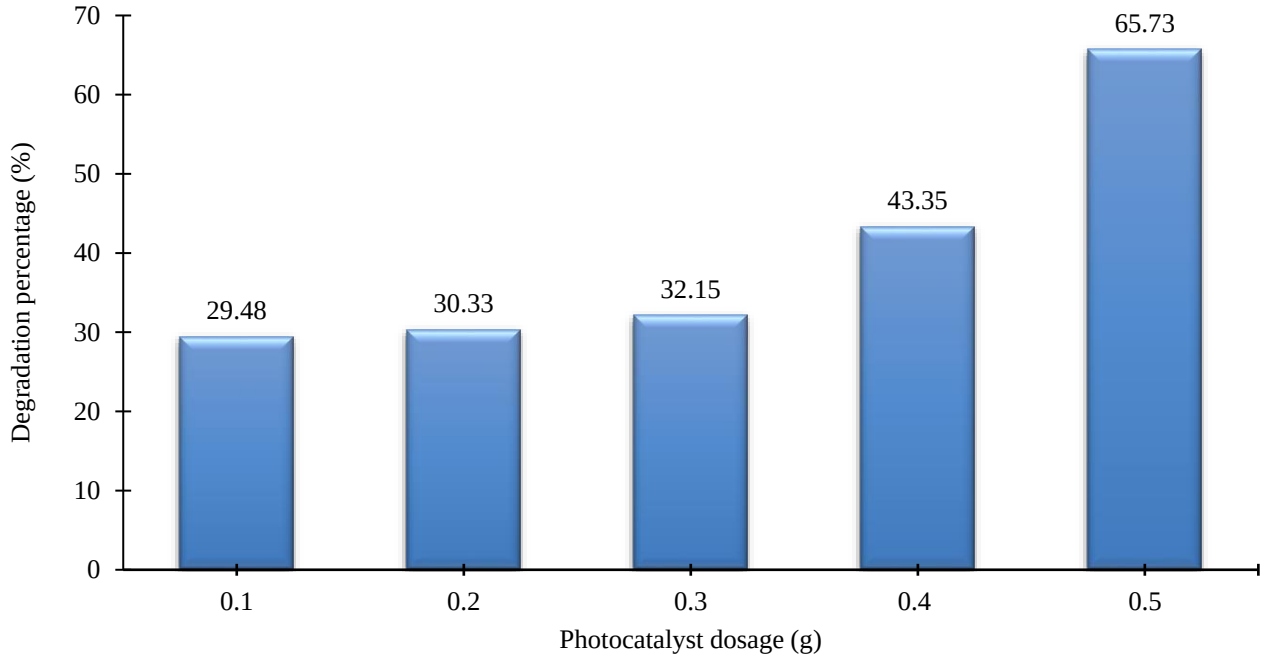

Figure 5. Degradation percentage of BPA with different photocatalyst dosages under visible light irradiation by using WO_3/ZnO (1:3) composite photocatalyst. Conditions: [BPA] = 10 mg/L, irradiation time = 180 min, pH = 2.

CONCLUSION

The WO_3/ZnO composite photocatalysts were successfully prepared via a simple chemical precipitation method. The highest degradation percentage of ~65% was obtained by 0.5 g of WO_3/ZnO (1:3) composite in degrading 10 mg/L of BPA in an aqueous solution. The improved photocatalytic activity may be attributed to the high dispersity of WO_3 on the surface of ZnO, effective charge separation of the photoexcited electrons and holes, and ease of electron migration. The best composite photocatalyst maintained its high degradation percentage for 180 min while remaining stable under 23 W of low-intensity visible light irradiation.

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AUTHOR CONTRIBUTIONS

Ts. Dr. Hamizah Mokhtar, ChM. Dr. Zul Adlan Mohd Hir, Dr. Hartini Ahmad Rafaie, and Ms. Nik Fatin Nabihah Atiqah Nik Ramli analyzed and evaluated the data to cater aims of this research and assist in writing for publication as well as writing the final report within the research scope.

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COMPETING INTEREST

The authors declare that there are no conflicts of interest associated with this publication.

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