



Photocatalytic Oxidation of Benzyl Alcohol to Benzyl Aldehyde Using Synthesized Zinc Oxide and Zinc-Doped Titanium Oxide

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Abstract: Photocatalytic oxidation of alcohols to carbonyl compounds is important in various organic transformations because it plays a significant role in synthesizing fine chemicals, confectionaries, fragrances, and pharmaceuticals. This study compares the photocatalytic activity of synthesised zinc oxide and zinc-doped titanium oxide for the photocatalytic oxidation of aromatic alcohols to aldehydes. We prepared undoped and zinc-doped titanium oxide (Zn-TiO₂) by hydrothermal synthesis. We evaluated the photocatalytic performance of the synthesised photocatalyst for converting benzyl alcohol to benzaldehyde. We used powder X-ray diffraction to characterise the synthesised product. Gas Chromatography - Flame Ionization Detector was employed to assess the photocatalytic conversion activity of Benzyl alcohol (BA) via UV-Visible irradiation. The study achieved about 40% conversion of the alcohol and 80% product selectivity. This research also compares hydrothermally synthesized Zinc oxide and Zn-TiO₂ as photocatalysts for the organic synthesis of aromatic hydrocarbons such as benzyl alcohol, where zinc-doped titanium oxide yields higher product selectivity than the undoped photocatalyst.

Keywords: Photocatalysis, Hydrothermal, Benzyl Alcohol, Benzyl Aldehyde, UV-visible irradiation.

Nomenclature:

BA: Benzyl Alcohol

UV: Ultraviolet

BAD: Benzyl Aldehyde

GC-FID: Gas Chromatography-Flame Ionization Detector

XRD: X-Ray Diffraction

NPs: Nanoparticles

ZnO: Zinc Oxide

AOP: Advanced Oxidation Process

I. INTRODUCTION

The selective oxidation of alcohols to carbonyl compounds represents a fundamental transformation in organic chemistry. Traditionally, this process has relied on hazardous, toxic stoichiometric oxidants and often required harsh conditions, such as elevated temperatures and pressures [4]. Ultraviolet- and visible-light-driven organic photochemical synthesis has recently emerged as a significant

area of research in academia and industry because it is environmentally friendly and sustainable. The visible spectrum is particularly advantageous for facilitating green organic photochemical reactions under catalytic conditions, given its abundance and sustainability [5]. The unique properties of photocatalysts have spurred research across diverse fields to advance green chemistry objectives [2]. Researchers synthesize photocatalysts in various media and use them in numerous applications, including water treatment, organic waste degradation, green photocatalytic oxy-functionalization of organic compounds, and selective organic transformations under environmentally benign conditions [9]. Zinc oxide (ZnO) nanoparticles, in particular, have found extensive applications in biosensors, gas sensors, solar cells, ceramics, nanogenerators, photodetectors, catalysts, ultraviolet (UV) adsorbents in cosmetics and veneers, antiviral agents, pigments, optical materials, and in electrical and optical processes [1].

Because aromatic aldehydes are used across a wide range of applications, they have drawn substantial commercial interest. Market estimates value the sector at approximately \$6.3 billion in 2022 and project growth to \$8.5 billion by 2031, at a compound annual growth rate of 3.9% [12]. Conventional aldehyde production frequently employs environmentally hazardous organic solvents and stoichiometric oxygen donors such as potassium permanganate (KMnO₄) and chromium trioxide (CrO₃) [7]. Because these reagents are toxic, costly, and generate significant waste, there is a critical need to develop more environmentally sustainable alternatives [14].

Photocatalysis is a bright technique for environmental remediation because it can degrade organic pollutants under lenient conditions. Several reports have employed matrices such as ZnO and TiO₂ as photocatalysts because of their high chemical stability, low toxicity, and low cost [11]. In these systems, the photocatalyst is the active material that enables light-induced degradation.

This research investigates selective oxidation reactions, which are important for green and sustainable chemistry because they minimize toxic waste generation. Photocatalytic oxidation, a type of advanced oxidation process (AOP), has attracted considerable interest because hydroxyl radicals effectively decompose organic pollutants [12]. In these processes, the catalyst generates electron-hole pairs under light irradiation, and these charge carriers facilitate pollutant conversion [13].

This study aims to evaluate the effectiveness of synthesized zinc oxide and zinc-doped titanium oxide as photocatalysts for converting benzyl alcohol to benzyl aldehyde.

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II. METHODOLOGY

A. Preparation of ZnO Nanoparticle

1.3 g zinc acetate was dissolved in 50 mL distilled water. Separately, 1g of NaOH was dissolved in 50 mL Ethanol. A magnetic Stirrer was used to ensure solution homogeneity. Then, NaOH_(aq) was added dropwise to the Zinc Acetate Solution and stirred for 1 h. The final mixture was transferred to an autoclave [10] and placed in an Oven at 95 °C for 2 h.

The mixture was then filtered, rinsed with distilled water, and dried in an oven at 60 °C [9]. The obtained powder was then annealed at 400 °C to improve the crystal structure. The whole process was repeated as required using 0.65g of Zinc Acetate, which was weighed into a 250mL beaker. The Powder was then characterized using XRD.

B. Preparation of ZnO-doped TiO₂ Nanoparticle

Hydrothermally Synthesised Zinc Oxide was mixed with Commercial TiO₂ powder at 0.1–1% ZnO. A magnetic Stirrer was used to ensure solution homogeneity. Then, NaOH_(aq) was added dropwise to the Solution, and the mixture was stirred for 1 h. The final mixture was transferred to an autoclave and placed in an Oven at 95 °C for 2 h [6]

The mixture was then filtered, rinsed with distilled water, and dried in an oven at 60 °C. The resulting powder was annealed at 400 °C.

C. Alcohol Conversion

i. Photocatalytic Conversion using Synthesized Zn-TiO₂

1g of the Synthesized catalyst (Zn-TiO₂) was placed in a 50 mL conical flask containing 100 mL of Distilled water as the solvent and 20 microliters of Benzyl alcohol, then placed in the UV chamber for 5 min, 10 min, 15 min, 20 min, 25 min, and 30 min. At each run, the process was repeated using 0.5 g, 1 g, 1.5 g, and 2 g of the photocatalyst [9]. The Results of each process at each time point were transferred into a clean amber reagent bottle for GC-FID analysis of the resulting product.

ii. Photocatalytic Conversion using Synthesized ZnO

0.051g of the Synthesized catalyst (ZnO) was placed in a 50 mL conical flask containing 100 mL of distilled water as the solvent and 20 microliters of BA, then placed in the UV chamber for 5 min, 10 min, 15 min, 20 min, 25 min, and 30 min. At each run, the process was repeated using 0.5 g, 1 g, 1.5 g, and 2 g of the photocatalyst. The Results of each process at each time point were transferred into a clean amber reagent bottle for GC-FID analysis of the resulting product.

III. RESULTS AND DISCUSSION

A. Synthesized Product Yield

Table 1 shows the yield of the synthesised ZnO and Zn-TiO₂ photocatalysts.

Table 1: Percentage Yield of the Synthesised ZnO and Zn-TiO₂

Sample	Weight (g)	Yield (%)
ZnO	0.182	23.40
Zn-TiO ₂	9.580	73.69

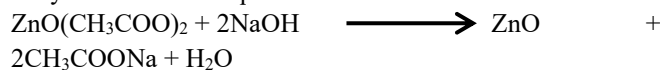
B. Percentage Yield

The formula for the expression of percentage yield of the synthesized product is given as:

$$\text{Yield (\%)} = \frac{\text{Weight of ZnO NPs}}{\text{Weight of C}_4\text{H}_6\text{O}_4\text{Zn}} \times 100$$

C. Calculation of Percentage Yield

We weighed the synthesised photocatalysts and recorded the yield. From the equation of the reaction below:

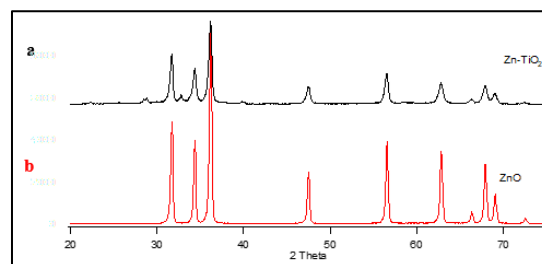


$$\begin{aligned} \text{Theoretical yield (g)} &= \text{moles} \times \text{molar mass} \\ &= 0.00953 \times 81.4 = 0.775\text{g} \end{aligned}$$

$$\begin{aligned} \% \text{ yield (\%)} &= \frac{\text{actual}}{\text{theoretical}} \times 100 \\ &= \frac{0.182}{0.775} \times 100 = 23.4\% \end{aligned}$$

As shown in Table I, the Product percentage yield of the synthesised ZnO is lower than that of the Zn-TiO₂ particle. The lower product percentage yield of hydrothermally synthesised ZnO nanoparticles than that of Zn-TiO₂ nanoparticles can be attributed to several factors, including stability, as the Zn-TiO₂ matrix is more stable, leading to a higher yield. Other factors include the catalyst, temperature, and other conditions.

D. Characterization of ZnO and Zn-TiO₂ Photocatalyst

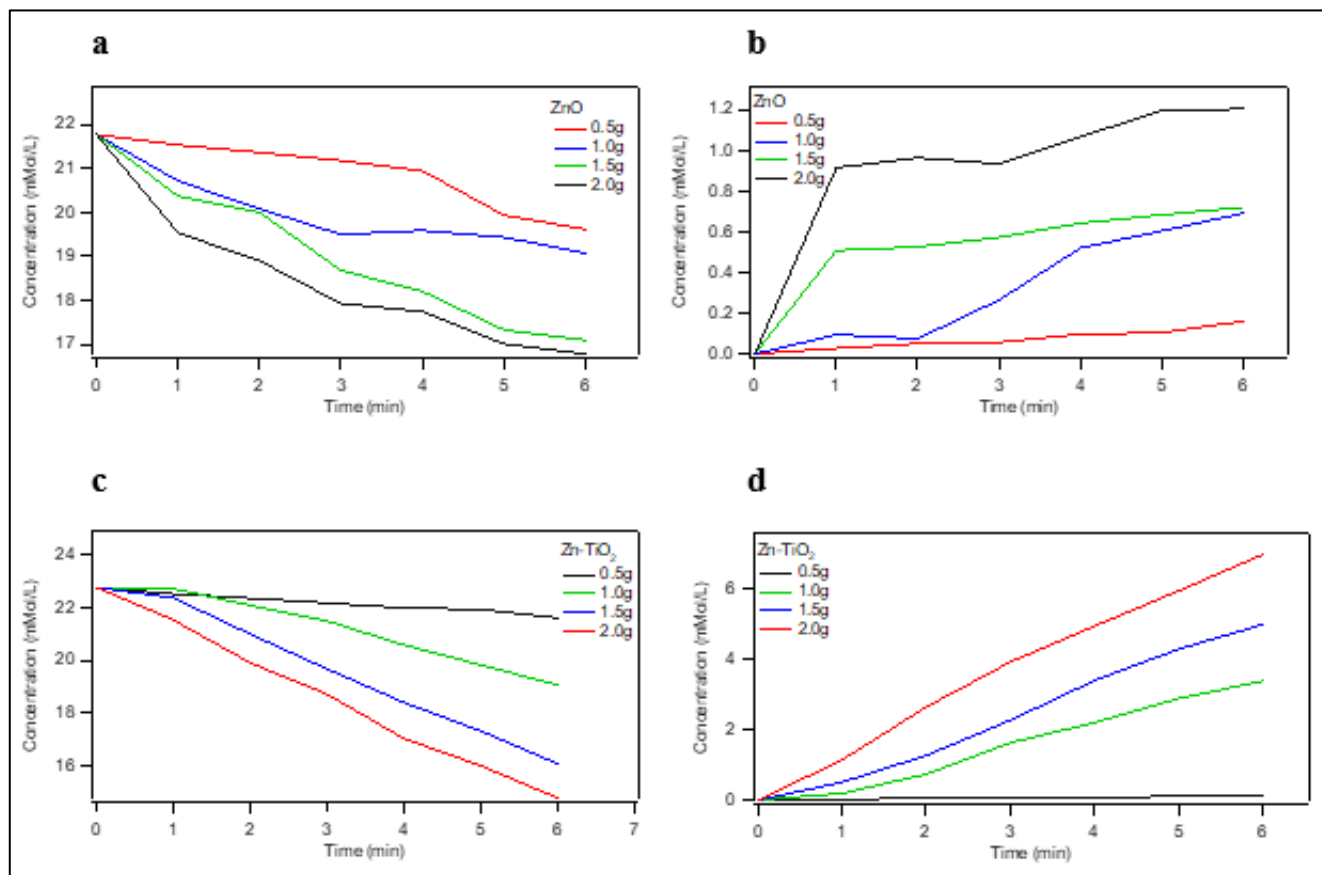


[Fig.1: X-ray Diffraction Spectra for (a). Zn-TiO₂ and (b). ZnO]

We used powder X-ray diffraction to characterise the synthesised products. We collected XRD patterns of all photocatalysts over $2\theta = 20^\circ - 70^\circ$ using an X-ray diffractometer with Cu-K α radiation.

Figure 1(b) and Figure 1(a) show the XRD patterns of the ZnO and Zn-TiO₂ photocatalysts, respectively. The XRD pattern of ZnO nanoparticles was uniform, with no impurities observed (Figure 1(a)). Moreover, the XRD pattern showed intense, sharp peaks, confirming the crystalline nature of the undoped ZnO nanoparticles. XRD peaks at 31.7, 34.35, and 36.15 confirmed the formation of a pure wurtzite structure in ZnO nanoparticles. The peak at 36.15 ° is the major peak, consistent with the hexagonal wurtzite structure [3]. The XRD results indicate good crystallinity in ZnO nanoparticles, as shown by the intense, narrow peaks.

Figure 1 (a) shows the XRD pattern of Zn-TiO₂ nanoparticles. The XRD pattern of Zn-TiO₂ nanoparticles was similar to that of ZnO nanoparticles. The XRD results showed narrow, sharp peaks, confirming the crystalline nature of the synthesized Zn-TiO₂ nanoparticles. This result aligns with [8]: higher crystallinity reduces defects that promote recombination of photoexcited electron-hole pairs and therefore improves the catalyst's overall photocatalytic activity.



[Fig.2: GC-FID Plot of Concentration (mMol/L) vs. Time (min) Showing the Effect of Catalyst Loading on (a). Photocatalytic Conversion of BA using ZnO (b). Production of BAD using ZnO (c). Photocatalytic Conversion of BA using Zn-TiO₂ (d). Production of BAD using Zn-TiO₂]

E. Photocatalytic Activity of the Synthesized Particles in Oxidation of Benzyl Alcohol to Benzaldehyde

The Photocatalytic activity of the synthesized ZnO and Zn-TiO₂ was studied using gas chromatography-flame ionization detection, with a plot of Concentration (mMol/L) vs. time (min). The experiment investigated the photocatalytic oxidation of benzyl alcohol to benzyl aldehyde using ZnO and Zn-TiO₂ nanoparticles as catalysts.

Figures 2a and 2c show the photocatalytic conversion of BA using ZnO and Zn-TiO₂, respectively, at catalyst loadings of 0.5 g, 1.0 g, 1.5 g, and 2 g.

As shown in Figure 2a, 0.5 g ZnO catalyst loading gave high BA conversion. BA converted at a steady rate in the initial 0–4 min, after which the plot showed a considerable decline as time increased. In contrast, at 2.0 g ZnO catalyst loading, BA conversion was rapid, and conversion continued over time. This implies that a ZnO catalyst loading of 2.0g shows high or preferential BA photocatalytic conversion.

In Figure 2c, a Zn-TiO₂ catalyst loading of 0.5g for BA conversion was less efficient, as the conversion rate was lower. In contrast, a Zn-TiO₂ catalyst loading of 2.0 g for BA conversion showed a sharp increase and reached a steady state over time. Thus, at 0.5 g Zn-TiO₂ catalyst loading, conversion is low, whereas at 2.0g, the BA Conversion rate is efficient.

Table II: Conversion/Selectivity Yield of Photo-Oxidation of BA to BAD using ZnO and Zn-TiO₂ Matrix

Sample	Conversion (%)	Selectivity (%)
ZnO	32.07	74.40
Zn-TiO ₂	40.25	80.04

F. Photocatalytic Activity of Synthesized Particles in the Oxidation of Benzyl Alcohol to Benzyl Aldehyde

The photocatalytic activity of synthesized ZnO and Zn-TiO₂ was evaluated using gas chromatography-flame ionization detection, with concentration (mMol/L) plotted against time (min). This experiment investigated the photocatalytic oxidation of benzyl alcohol to benzyl aldehyde using ZnO and Zn-TiO₂ nanoparticles as catalysts.

Figures 2a and 2c present the photocatalytic conversion of BA using ZnO and Zn-TiO₂, respectively, at catalyst loadings of 0.5 g, 1.0 g, 1.5 g, and 2.0 g.

Figure 2a shows that a 0.5 g ZnO catalyst loading resulted in a high initial BA concentration, with conversion occurring at a steady rate from 0 to 4 minutes, followed by a significant decline over time. In contrast, at a 2.0 g ZnO catalyst loading, BA conversion proceeded rapidly and completed more efficiently. These results indicate that a 2.0 g ZnO catalyst loading provides superior photocatalytic BA conversion.

As shown in Figure 2c, a 0.5 g Zn-TiO₂ catalyst loading resulted in a low BA conversion rate. In contrast, a 2.0 g Zn-TiO₂ catalyst loading produced a sharp and steady increase in BA conversion over time. Therefore, a 2.0 g Zn-TiO₂ catalyst loading shows a more efficient BA conversion rate than the 0.5g loading.

IV. CONCLUSION

Although TiO₂ has been thoroughly investigated as an effective photocatalyst, ZnO



is a suitable alternative because it is less expensive and has a similar band gap (~3.3 eV). Zn-TiO₂ has been reported to achieve higher oxidation rates (percentage conversion) and BA selectivity because it has higher photoexcitation efficiency under UV-visible light. Furthermore, hydrothermal synthesis of ZnO and Zn-TiO₂ provided a suitable, cost-effective route; XRD confirmed that the resulting materials were crystalline and had a wurtzite structure. To further improve photocatalytic activity, the catalyst surface area needs to increase to enable optimal mass transfer and efficient dye degradation, without adding equipment costs for separating the catalyst from water.

DECLARATION STATEMENT

Some of the cited references are older and are noted explicitly as [8]. However, these works remain significant for the current study, as they are pioneering in their fields.

After aggregating input from all authors, I must verify the accuracy of the following information as the article's author.

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