



## Comparative Study of Physical and Chemical Methods for Synthesizing Zinc Oxide - Loaded Polypropylene Non-Woven Fabrics for Photocatalytic Degradation of Benzophenone-3.

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### ABSTRACT

Benzophenone-3 (BP-3) is a well-known UV filtering ingredient in chemical sunscreens and other personal care products. However, high levels of BP-3 in discharged sewage water streams have been linked to reproductive damage, abnormal fetal development, endocrine system disruption, and neurotoxicity in experimental animal models. In this study, zinc oxide (ZnO) loaded on polypropylene non-woven fabrics (PNF) as a photocatalyst was created using two different approaches: the physical method and the chemical method. For physical method, ZnO was loaded onto PNF without addition of other chemical while for chemical method, Iminodiacetic acid (IDA) was added as a polymer ligand to hold onto the connection between PNF and ZnO, which helps improve the efficiency and stability of the synthesis process. The study found that PNF/Zn-IDA which is chemically synthesized was effective in degrading BP-3 by up to 93.38% compared to physically synthesized PNF/ZnO which can only remove 18.46% of BP-3. This approach offers a promising solution for the removal of hazardous chemicals from wastewater streams, contributing to a safer and more sustainable environment.

## 1. Introduction

The sun emits three ultraviolet (UV) radiation types - UVA, UVB, and UVC. Humans and other organisms living on Earth need an adequate supply of sunlight to live healthily. UVB reaches human skin and interacts with the 7-DHC protein to produce vitamin D, which promotes bone health, lowers blood pressure, and reduces inflammation. However, excessive exposure to UV radiation can have negative health impacts. For example, UVA radiation can prematurely age skin, damage eyes, and weaken the immune system, reducing disease resistance. It raises skin cancer risk. Overexposure to UVB can cause sunburn, skin cancer, and snow blindness [1].

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To avoid UV effect, humans usually minimize their time for noon outdoor activities, wear protective garments, seek shade, and use lots of sunscreens. Physical and chemical sunscreens contain many UV rays and help reduce UV exposure to human skin. Benzophenone-3 (BP-3) is a well-known UV filtering ingredient in chemical (organic) sunscreens and other personal care products such as shampoo, conditioner, deodorant, etc. BP-3, also called 2-hydroxy-4-methoxybenzophenone, or oxybenzone, is a lipophilic organic molecule having a molecular weight of 228.25 g/mol. The formulations of BP-3 themselves may have an aromatic structure that could absorb and stabilize the UV light that reaches the skin [2].

Despite its extensive use, BP-3 is present in high amounts in discharged sewage water streams, destroying the reproductive organs of humans and some aquatic life [3, 4]. Following a single application of sunscreen, the body excretes only 0.5% of the BP-3 compound that enters the bloodstream through urine within an extended period of 48 hours. Significantly, BP-3 can easily build up in adipose tissue because of its strong affinity to lipids [5]. It also has been confirmed that BP-3 can pass through the bloodstream, the blood-brain barrier, and the blood-placental barrier. Consequently, it possesses the capacity to induce harm to the reproductive system, lead to improper development of fetuses, alter the endocrine system, and cause neurotoxic effects in animal models used for experimentation. [2]. The phenol compounds identified in BP-3 are also contributing to poor birth outcomes. This is because they disrupt the endocrine system, which is caused by a change in the activity system of the sex hormone during crucial phases of development [6]. In animal experiments, biotransformation of BP-3 into hydroxylated forms was demonstrated, with some of these forms having higher estrogenic affinity both in vivo and in vitro. The persistent release of this set of chemicals into the ocean, as well as their ability to accumulate in living things, may have long-term repercussions on marine life [1].

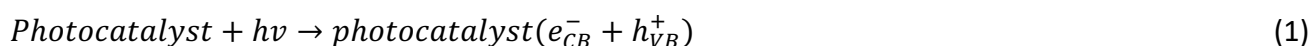
As a result, BP-3 must be handled with extreme caution to keep aquatic life's reproductive systems remaining intact. Degradation-based technologies, such as chemical oxidation, chlorination, and advanced oxidation processes (AOPs), could be used to completely remove or degrade various types of UV filters [7]. According to Ricardo *et. al*, a few researchers concluded that AOPs can be used to degrade BP-3 either individually or mixed with other pollutants in aqueous environmental matrices [8]. Removal of BP-3 using chlorination, chemical oxidation, and AOPs can mostly be done up to 78% and above. However, AOPs have gained popularity due to their effectiveness in pollutant degradation [5, 9]. One of the AOPs, photocatalysis, is a promising AOP that helps to mineralize pollutants using a photocatalyst. Photocatalyst dope or immobilization with other materials can be employed in photocatalytic AOPs to increase degrading efficiency [10].

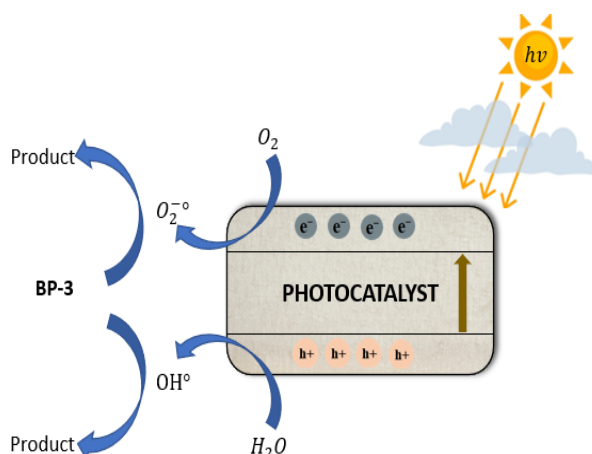
### 1.1 Principles and mechanism for photocatalytic degradation

There are several steps for photocatalytic degradation to occur. Photocatalysts need to be activated first by being illuminated with UV radiation [11]. The overall scheme of photocatalysis applied for the degradation of BP-3 is shown in Figure 1.

#### a. Photon excitation.

During irradiation, the photon from UV ( $h\nu$ ) cause the photoelectron from the valence band (VB) to excite and pass into the conduction band (CB). This process produces a positive hole ( $h_{VB}^+$ ) in VB and photo-induced electron ( $e_{CB}^-$ ) in CB. As a result, electron-hole pair ( $e^-/h^+$ ) are also formed. The Eq. (1) below indicates the reactions involved.





**Fig. 1.** Mechanism of photocatalytic degradation of the pollutants compound

b. Reaction with moisture.

The  $h_{VB}^+$  reacts with moisture ( $H_2O$ ), producing hydroxyl radicals ( $OH^\circ$ ). This  $OH^\circ$  acts as a powerful oxidizing agent, attacking most organic compounds and producing new non-hazardous chemicals as shown in Eq. (2) below:



c. Superoxide anion radical formation.

During the reaction of  $h_{VB}^+$  with  $H_2O$ , the  $e_{CB}^-$  in CB will react with oxygen from surroundings and generate superoxide anion radical ( $O_2^{-\circ}$ ) as shown in Eq. (3) below:



d. Degradation process

Both  $OH^\circ$  and  $O_2^{-\circ}$  will mineralize the pollutants into most stable non-hazardous substances as in Eq. (4) and Eq. (5) below:



However, the photocatalytic degradation itself depends on catalyst type and composition, light intensity, pollutant type and concentration, catalyst dosage, and solution pH.

1.2 New advanced photocatalyst

In photocatalytic degradation processes, several photocatalysts, such as titanium dioxide ( $TiO_2$ ), zinc oxide ( $ZnO$ ), tungsten trioxide ( $WO_3$ ), vanadium (V) oxide ( $V_2O_5$ ), iron (III) oxide ( $Fe_2O_3$ ), niobium pentoxide ( $Nb_2O_5$ ), and zirconia ( $ZrO_2$ ), have a positive outcome for photocatalytic reactions [12]. However,  $ZnO$  is one of the most promising photocatalysts for eliminating pollutants from wastewater due to its exceptional features. Over many years,  $ZnO$  has been used in a variety of applications, including cosmetics, medicine, electronics, and as a catalyst, particularly in

photocatalytic degradation. ZnO was regarded as an optimistic catalyst for resolving environmental problems because of its excellent electrical capabilities, high stability, good thermal transfer, and broad surface area [13, 14].

Nowadays, an improvised photocatalyst that is metal loaded on fabric is extensively employed in photocatalytic degradation because it helps to overcome with powdered photocatalysts that are difficult to recover, recycle, and separate and may pose issues for the environment and health [15]. For instance, different types of metal were synthesized and physically loaded onto fabric using simple dip-coat technique by Soonhyun *et al.* [16], Jatinder & Bansal [17], Ait-Touchente *et al.* [18], Cheng *et al.* [19], Zhu *et al.* [20], and Nozari *et al.* [21]. The photocatalyst that was synthesized included plastic optical fiber fabric coated with titanium dioxide (POF/TiO<sub>2</sub>), TiO<sub>2</sub>/cotton fabric, and CuFe<sub>2</sub>O<sub>4</sub> onto ceramic fabric, ZnO-NP-coated cotton fibres, (ZnO@SiO<sub>2</sub> NPs) catalyst on polyester fabric (PET) to degrade different types of dyes. Every study demonstrates a greater percentage of degradation, reaching 80% and beyond [16, 17, 18, 19, 20, 21].

Even though an improvised photocatalyst has been successfully synthesized, additional issues have been brought up, including leaching readily because there isn't sufficient support to sustain the catalyst attached to the fabric and uncontrolled agglomeration that will hinder the catalyst's effectiveness and reduce its stability [22]. To solve the problems, a new, more sophisticated photocatalyst will be created using chemical approaches. Iminodiacetic acid will be employed as a polymer ligand exchanger to hold onto the connection between fabrics and metals, strengthening the photocatalyst's stabilization. By using the grafting-from technique, IDA will form covalent bonds with the metal surface and the polymer layer or fabric [23]. It is commonly recognised that IDA's functional group has a strong affinity for a variety of metal ions, such as zinc, copper, and nickel [24]. As IDA is an amino dicarboxylic acid, it is very effective in controlling the development of nanoparticles during synthesis and maintaining the stability of the particles in solution [25].

This study compares the physical and chemical methods for synthesizing ZnO onto PNF to degrade BP-3 using photocatalytic degradation. The incorporation of IDA into the chemically synthesized photocatalyst enhances its efficiency and stability, providing a novel advanced photocatalyst with improved performance.

## 2. Methodology

### 2.1 Materials

Glycidyl Methacrylate (GMA) grafted-PNF was supplied by obtained from Agensi Nukleor Malaysia. IDA was supplied by Sigma-Aldrich. Acetic acid (CH<sub>3</sub>COOH), glycerol (C<sub>3</sub>H<sub>8</sub>O<sub>3</sub>), potassium hydroxide (KOH), sodium hydroxide (NaOH), and zinc acetate (Zn(CH<sub>3</sub>CO<sub>2</sub>)<sub>2</sub>) were supplied by R&M Chemicals while ethanol (C<sub>2</sub>H<sub>5</sub>OH) were purchased from Hmbg Chemicals. All solutions were prepared by using deionized water produced from unknown.

### 2.2 Physical method in preparation of ZnO loaded GMA grafted-PNF (PNF/ZnO)

The PNF was loaded with 0.7 M Zn(CH<sub>3</sub>CO<sub>2</sub>)<sub>2</sub> solution, by immersed it inside the hydrothermal autoclave. Then, the hydrothermal autoclave was put in a vacuum oven at 80 °C for 24 hours. This is followed by several washed in deionized water and an overnight drying period in a vacuum oven at 60°C. The solution containing C<sub>2</sub>H<sub>5</sub>OH/deionized water/ C<sub>3</sub>H<sub>8</sub>O<sub>3</sub> (40/40/20 (v/v)) was prepared, and 1.0 M of KOH solution was added drop by drop while being continuously stirred until pH 12 is reached. The Zn-loaded sample and the solution prepared were combined and poured inside the hydrothermal autoclave. The hydrothermal autoclave was then put in a vacuum oven for an overnight at 80 °C.

After that, the sample was washed several times in deionized water and dried in a vacuum oven at 60 °C.

### 2.3 Chemical method in preparation of ZnO loaded IDA in GMA grafted-polypropylene non-woven fabric (PNF/Zn-IDA)

First, an C<sub>2</sub>H<sub>5</sub>OH/deionized water (50/50 (v/v)) solution containing 0.2 M IDA was used to soak the GMA grafted-PNF. The reaction took place inside the hydrothermal autoclave at 80°C. The remaining unreacted epoxide groups were hydrolyzed with 0.5 M CH<sub>3</sub>COOH for an hour at 80°C inside the hydrothermal autoclave after IDA groups were bounded to the epoxy group of the grafted polymer branches. Then, the sample was reacted with 1.0 M NaOH inside the hydrothermal autoclave in a vacuum oven at 80°C for half an hour and washed in deionized water and dried twice: initially at ambient temperature and then at 60°C in a vacuum oven.

The sample containing Na-substituted IDA was loaded with 0.7 M Zn(CH<sub>3</sub>CO<sub>2</sub>)<sub>2</sub> solution by immersing it in the hydrothermal autoclave. Then, the hydrothermal autoclave was put in a vacuum oven at 80 °C for 24 hours. This is followed by several washes in deionized water and an overnight drying period in a vacuum oven at 60°C. The solution containing C<sub>2</sub>H<sub>5</sub>OH/deionized water/ C<sub>3</sub>H<sub>8</sub>O<sub>3</sub> (40/40/20 (v/v)) was prepared, and 1.0 M of KOH solution was added drop by drop while being continuously stirred until pH 12 is reached. The Zn-loaded sample and the solution prepared were combined and poured inside the hydrothermal autoclave. The hydrothermal autoclave was then put in a vacuum oven for an overnight at 80 °C. After that, the sample was washed several times in deionized water and dried in a vacuum oven at 60 °C.

### 2.4 Photocatalytic Degradation Performances of BP-3

The photocatalytic testing of PNF/ZnO photocatalyst was evaluated by degradation of BP-3 in a batch flow reactor as in Figure 1. The PNF/ZnO photocatalyst was inserted into 100 mL of 1ppm BP-3 solution inside the reactor. The solution was stirred first in a dark condition for 30 minutes to achieve adsorption-desorption equilibrium [7]. Then, the mercury lamp with an intensity of 9W (UV light) was on to supply the irradiation with a wavelength of 254 nm for 300 minutes. The solution was collected around 4mL in certain time intervals to analyze the maximum absorbance of BP-3 at 256nm by UV-Vis spectrophotometer. The same procedure was used to test for photocatalytic degradation using PNF/Zn-IDA photocatalyst. To ensure the accuracy of the obtained data, the same experiment was conducted in triplicate. The percentage removal of BP-3 can be calculated using Eq. (6) below:

$$\text{Percentage removal (\%)} = \frac{C_0 - C_f}{C_0} \times 100 \quad (6)$$

Where  $C_0$  stands for the initial concentration of BP-3 and  $C_f$  stands for the final concentration of BP-3. Figure 2 illustrates the diagram of the batch flow photocatalytic reactor that is used in this study.



**Fig. 2** Batch Flow Photocatalytic Reactor Diagram

### 3. Results

Figure 3 illustrates the time-dependent photocatalytic degradation of BP-3 using PNF/ZnO and PNF/Zn-IDA. Before exposure to UV radiation, the catalyst was stirred continuously with BP-3 under dark conditions for 30 minutes to achieve adsorption-desorption equilibrium. The results show that PNF/ZnO exhibited minimal adsorption, whereas PNF/Zn-IDA adsorbed approximately 3% of BP-3 under dark conditions. Notably, all catalysts demonstrated improved performance under UV-light irradiation compared to dark conditions [26]. PNF/ZnO degraded BP-3 at a slow rate, achieving 18.5% removal after 300 minutes of UV irradiation. The lower removal percentage observed with PNF/ZnO could be attributed to the instability of the photocatalyst itself. This instability led to agglomeration, which in turn compromised its ability to effectively degrade BP-3 [22]. Additionally, the use of a physical method to synthesize PNF/ZnO can potentially cause the formation of cracks on fabrics. This is primarily due to the shrinkage of the precipitate, which is a consequence of thermal decomposition [27]. In contrast, PNF/Zn-IDA showed a significant degradation of BP-3, reaching 92.6% removal within the same duration. This suggests that the addition of IDA to the catalyst through the chemical method enhances photocatalytic degradation. This enhancement is likely due to the interaction between Zn ions and IDA, which might improve the band structure, optical, and electrical properties of the catalyst. Furthermore, the addition of IDA also helps the deposition of powder on fabrics by covalently binding to the Zn through a chemical reaction by connecting the functional end group of IDA with the Zn [22].

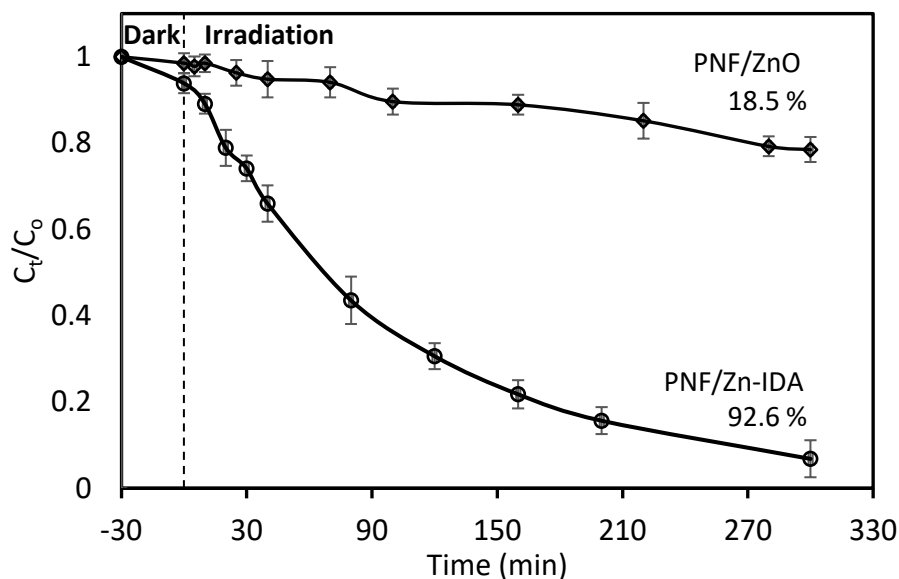


Fig. 3 Photocatalytic degradation percentage of PNF/ZnO and PNF/Zn-IDA

#### 4. Conclusions

In this study, PNF/ZnO was successfully synthesized using a physical dip-coating method and PNF/Zn-IDA through a chemical process. The photocatalytic degradation of BP-3 occurred within 300 minutes of UV radiation exposure. Notably, the use of IDA as a polymer ligand to maintain the bond between PNF and ZnO resulted in a significant improvement in the degradation of BP-3, with a removal rate of up to 92.6% compared to PNF/ZnO, which only removed 18.5% of BP-3. These findings suggest a novel approach to developing enhanced photocatalysts, particularly for the degradation of cosmetic chemical compounds like BP-3.

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