FREE STANDING TITANIUM DIOXIDE HOLLOW NANOFIBERS FOR PHOTOCATALYTIC DEGRADATION OF BISPHENOL A

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ABSTRACT

Modernization and urbanization have adversely affected water quality and harmed the sustainability of water sources. Bisphenol A (BPA) has been identified as an endocrine-disrupting compound that, when exposed to the human body, can interfere with the hormone system and cause severe health effects and disorders. Titanium dioxide (TiO_2), a prevalently used semiconductor in photocatalytic degradation fields, has wide bandgap energy and a low specific surface area. These properties can lead to a decline in photocatalytic degradation performance. The template synthesis approach can be used to produce hollow nanofibers photocatalysts with a large surface area, a narrow bandgap, and excellent degradation capability. This process, however, yields powder-form photocatalysts that require post-recovery treatment before being recycled in a photocatalytic slurry system. In this study, TiO_2 hollow nanofibers (THNFs) were developed at various calcination temperatures. THNFs produced at 600 °C (THNF600) produced nanofibers with the best hollow morphology, with a bandgap of 3.0 eV, with a specific surface area of 81.2776 m^2/g, and mixed-phase of 24.2 % anatase and 75.8 % rutile. As a result of the large surface area and excellent optical properties, the THNFs exhibited the highest BPA degradation of 71.48%. This result was also significantly better than that of Degussa P25, a commercial TiO_2, with BPA degrades at only 38.62%. Using THNF600, the optimum photocatalysts dosage, pH, and initial BPA concentration were determined to be 0.75 g/L, pH 4.1, and 10 ppm, respectively. Then, the powder-form THNF600 was assembled into a free-standing form using chemical treatment and vacuum filtration technique. Free-standing THNFs containing 0.75 g of THNF600 (FS75-THNFs) exhibited good adherence and connectivity between the nanofibers. After five cycles of reaction, the THNF600 experienced an average of 14.38% catalyst loss. The recyclability of FS75-THNFs outperformed the THNF600 which gave 5% average catalyst loss from its original weight while maintaining excellent degradation performance. In conclusion, this study recommends the potential application of free-standing TiO_2 hollow nanofibers as the high potential novel photocatalysts for the treatment of BPA in wastewater.

ABSTRAK

Pemodenan dan pembandaran telah memberi kesan yang buruk ke atas kualiti air dan merosakkan kelangsungan bekalan air. Bisphenol A (BPA) telah dikesan sebagai sebatian pengganggu endokrin yang boleh mengganggu sistem hormon dan memberi kesan buruk terhadap kesihatan badan manusia. Titanium dioksida (TiO_2), semikonduktor yang digunakan secara meluas di dalam bidang penguraian fotobermangkin, mempunyai jurang jalur tenaga yang luas dan luas permukaan yang rendah. Sifat-sifat ini boleh menyebabkan penurunan dalam prestasi penguraian fotobermangkin. Teknik sintesis bertemplat boleh digunakan untuk menghasilkan fotomangkin gentian nano geronggang yang mempunyai luas permukaan yang besar, jurang jalur tenaga yang kecil, dan kemampuan penguraian yang cemerlang. Walaubagaimanapun, proses ini menghasilkan fotomangkin berbentuk serbuk yang memerlukan rawatan pasca-pemulihan sebelum dikitar semula di dalam sistem fotobermangkin buburan. Dalam kajian ini, penghasilan gentian nano geronggang titanium dioksida (THNFs) dilakukan pada suhu pengkalsinan yang berbeza. THNFs yang dihasilkan pada suhu 600 °C (THNF600) menghasilkan gentian nano dengan morfologi geronggang yang terbaik, dengan jurang jalur tenaga sebanyak 3.0 eV, luas permukaan 81.2776 m^2/g, dan campuran fasa sebanyak 24.2 % anatase dan 75.8 % rutil. Disebabkan oleh luas permukaan yang besar dan ciri optikal yang baik, dengan THNFs tersebut menunjukkan penguraian BPA yang tertinggi sebanyak 71.48%. Keputusan ini juga lebih baik dari pencapaian Degussa P25, sejenis TiO_2 komersial yang menunjukkan penguraian BPA hanya 38.62%. Menggunakan gentian THNF600, dos fotomangkin, nilai pH, dan kepekatan awal BPA yang optima telah ditentukan masing-masing pada 0.75 g/L, pH 4.1 dan 10 ppm. Kemudian, THNF600 berbentuk serbuk dihimpunkan dalam bentuk mandiri menggunakan rawatan kimia dan penurasan vakum. THNFs mandiri yang mempunyai 0.75 g THNF600 (FS75-THNFs) menunjukkan perekatan dan penyambungan yang baik antara gentian-gentian nano. Selepas lima kitaran tindak balas, THNF600 mengalami purata kehilangan fotomangkin sebanyak 14.38%. Kebolehkitaran semula FS75-THNFs menandingi THNF600 dengan purata kehilangan fotomangkin sebanyak 5% dan mengekalkan pencapaian penguraian yang cemerlang. Kesimpulannya, gentian nano geronggang TiO_2 mandiri mempunyai potensi yang tinggi sebagai fotomangkin baharu bagi rawatan BPA dalam air sisa.

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LIST OF ABBREVIATIONS

BPA	-	Bisphenol A
CB	-	Conduction band
DDT	-	Dichlorodiphenyltrichloroethane
DMF	-	N,N-dimethylformamide
DOE	-	Department of Environment
EDC	-	Endocrine-disrupting compound
EDX	-	Energy dispersive x-ray
FESEM	-	Field emission scanning electron microscopy
FS-THNFs	-	Free-standing titanium dioxide hollow nanofibers
FTIR	-	Fourier-transform infrared
HPLC	-	High-performance liquid chromatography
PAN	-	Polyacrylonitrile
PCBs	-	Polychlorinated biphenyls
REACH	-	Registration, Evaluation, Authorisation, and Restriction of Chemicals
TGA	-	Thermogravimetric analysis
THNFs	-	Titanium dioxide hollow nanofibers
TiO_2	-	Titanium dioxide
TTIP	-	Titanium (IV) isopropoxide
USEPA	-	United States Environmental Protection Agency
UV	-	Ultraviolet
VB	-	Valence band
XRD	-	X-ray diffractometer

LIST OF SYMBOLS

°C	-	Degree celcius
e^-	-	Electrons
h^+	-	Holes
S_{BET}	-	Specific surface area
C_0	-	Initial BPA concentration
C_t	-	BPA concentration at time t
g/L	-	Gram per litre
nm	-	Nanometre
ppm	-	Part per million

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

A clean water supply has become vital in our daily lives since water is required for drinking, cooking, cleaning, and other daily activities. Water should be clean and devoid of hazardous chemicals that might cause future health concerns in the short or long term. However, increased industrialization, rising population, and climate change have made it difficult to provide sufficient clean and safe water sources. According to Liao *et al.* (2021), by 2050, the global water demand will increase by 20-30% due to increasing industrial and domestic water use. In Malaysia, modernization and urbanization have caused water pollution and have an adverse effect on the sustainability of water sources. The Department of Environment (DOE) has classified most of Malaysia's river water quality is in Water Quality Index Class II and Class III (Afroz and Rahman, 2017). This classification implies that these water sources need to undergo some conventional and extensive treatment before it is safe to be consumed. Wastewater can be defined as contaminated and used water from residential, domestic, commercial, industrial, and agricultural activities. Wastewater contains microorganisms, nutrients, metals, and emerging contaminants. One of the compounds that is identified as the emerging contaminant and ubiquitously found in wastewater is Bisphenol A (BPA).

BPA is mainly used as an intermediate in polycarbonate plastics and epoxy resins production. BPA is used in many sectors, including producing CDs and DVDs, electrical equipment, vehicles, construction glazing, sports safety equipment, medical devices, dinnerware, baby bottles, and food storage containers. Meanwhile, epoxy resins are utilized in the internal coating to prevent food and beverages from directly contacting metals (Huang *et al.*, 2012). Despite its usage as a primary material in many manufacturing industries, BPA is an endocrine-disrupting compound (EDC) that, at certain doses, can interfere with the hormone system. If exposed to the human body, it can induce severe health effects and disorders. As a result, it is critical to remove BPA from wastewater before being used. Photocatalytic degradation technology has been introduced to remove the hazardous compounds in wastewater. Via photocatalytic process, BPA can be degraded into less harmful species such as carbon dioxide and water under the presence of light. This technology offers great advantages such as a wide range of organic pollutants that can be mineralized, minimum use of chemicals, and not involving sludge production and disposal at the end of the process (Molinari *et al.*, 2017). Metal oxides such as titanium dioxide (TiO_2) is suitable to be used as photocatalyst because of their strong oxidizing abilities for the decomposition of organic pollutants, high hydrophilicity, chemical stability, long durability, nontoxicity, low cost, and transparency to visible light (Nakata and Fujishima, 2012). The commercial TiO_2 (Degussa P25) is commonly used as a photocatalyst. However, Degussa P25 is known for its low reported specific surface area (50 m^2/g) and wide bandgap energy (3.2 eV) (Uddin *et al.*, 2020). Therefore, morphology control of TiO_2 photocatalysts is necessary to produce a photocatalytic material with superior specific surface area and narrow bandgap energy.

The emerging discovery of hollow nanostructures has motivated us to synthesize TiO_2 hollow nanofibers (THNFs) for BPA photodegradation via template synthesis technique. According to Chu *et al.* (2012), hollow nanofibers have a very extensive surface area favorable for surface-related applications like photocatalytic reactions. Other than that, the tubular shape of hollow nanofibers provides optimum physicochemical properties for electron transportation. A study reported that the surface area of hollow nanofibers is about twice larger than that of conventional nanofibers (Wang *et al.*, 2016b). In this study, the morphology and optical properties of the synthesized photocatalysts were controlled by investigating the effect of varying calcination temperatures from 400 to 600 °C. Apart from the morphology, the recyclability of the photocatalysts is also essential in determining the overall performance of the photocatalysts.

Powder-formed photocatalysts are often used in a photocatalytic slurry system, which bears the risk of experiencing catalyst loss at the end of the reaction. On the other hand, photocatalysts that are immobilized into a membrane or a substrate suffer from the hindered active surface area. Hence, we explored the potential of assembling the synthesized THNFs into a free-standing structure (FS-THNFs) film via facile chemical treatment and vacuum filtration. The FS-THNFs are firmly adhered and have enhanced recyclability properties while maintaining the high photodegradation performance throughout repeated reaction cycles.

1.2 Problem Statement

The presence of endocrine disruptors in wastewater has become a concern because of the health effects that they cause. One of the identified endocrine disruptors is Bisphenol A (BPA), which is used as the monomer in numerous manufacturing industries such as food containers, plastic bottles, food can sealants, and electronic equipment. Previous studies reported that the entrance of BPA into the human body could cause reproductive problems, cardiovascular diseases, and cancerous tumors.

Due to the high resistance of organic pollutants, existing conventional treatments to clean-up water often fail to treat these contaminants, resulting in high concentrations discharged in the treated effluents. For instance, filtration, adsorption, and chemical oxidation do not destroy the pollutants but produce suspended particles or sludge that require post-treatment disposal. The pollutants can adsorb onto the surface of suspended solids that were not completely removed in the conventional water treatment. Photocatalytic degradation has been gaining attention for its ability to oxidize a wide range of organic compounds employing semiconductor photocatalysts. The commercially available TiO_2 photocatalyst, Degussa P25, is advantageous due to its efficient photoactivity, low cost, high stability, and non-toxicity.

However, since the main fraction of Degussa P25 is anatase, it has a large bandgap, which is 3.2 eV. Photocatalysts with a larger bandgap require higher energy to be activated to initiate photodegradation of pollutants. Besides the large bandgap, Degussa P25 also has a low surface area of only ~50 m^2/g, limiting the available UV irradiation sites. This inhibits the production of hydroxyl radicals to attack the organic pollutants molecules.

Hollow nanostructured TiO_2 with high surface area, low bandgap energy, and superior photocatalytic performance can be synthesized via template synthesis, which involves a calcination process at high temperature. As a result, the synthesized photocatalysts are often present in powder form and used in the slurry photocatalytic systems. Despite providing increased exposure to UV light, the remaining pitfall of this system is the low recyclability of photocatalysts, which are often trapped in the effluent. The catalysts loss issue causes reduced photocatalytic performance, besides increasing the operational cost of the process, especially in a larger scale set-up. The development of an immobilized photocatalytic membrane has been studied for the past few years to eliminate the separation and post-recovery process of small-sized catalysts. However, the immobilized photocatalysts were found to reduce efficiency because of the smaller contact area with the pollutants.

Therefore, this study aims to overcome the challenges mentioned above via the development of free-standing TiO_2 hollow nanofibers, as illustrated in Figure 1.1. The THNFs with the best performance from varied calcination temperatures are assembled into a free-standing form via chemical treatment followed by vacuum filtration. In this step, determining the minimum amount of THNFs is essential to optimize the processing cost while maintaining the photocatalytic ability, ensuring the excellent recyclability of the photocatalysts and eliminating the inconvenient catalyst recovery steps in the existing system. Although many scientific journal papers are published each year and significant progress has been made in recent years, there is still a lack of comprehensive research studies that aim to close the gap on the mechanistic insights of the formation of THNFs using template synthesis technique and the application of free-standing THNFs for the photocatalytic oxidation of BPA.

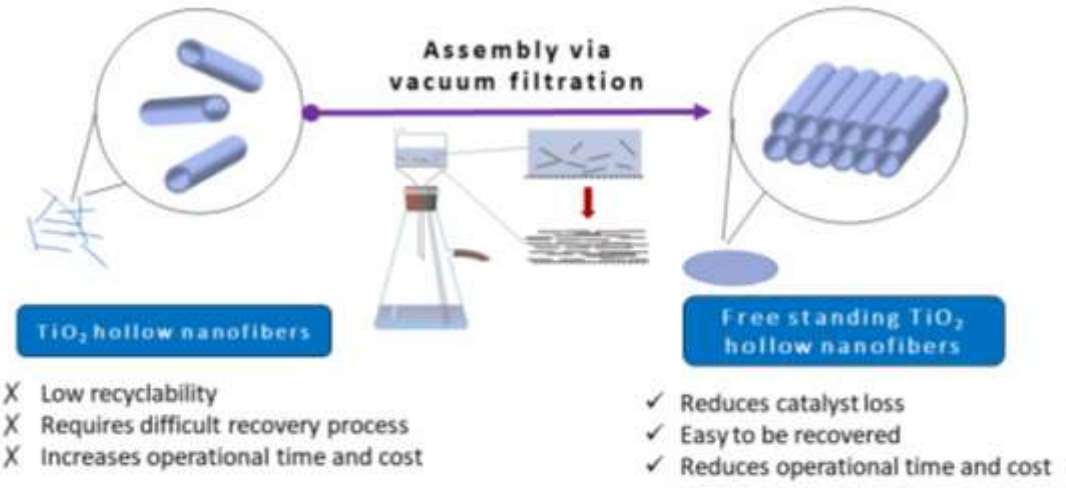

Figure 1.1 Comparison between hollow nanofibers and free-standing hollow nanofibers

1.3 Objective of Study

1. To determine the effect of calcination temperature on the formation of THNFs, the physicochemical properties, and photocatalytic performance

2. To examine the photocatalytic performance of THNFs on BPA as a function of THNFs dosage, pH of BPA solution, and initial concentration of BPA solution in comparison to Degussa P25 TiO_2

3. To synthesize free-standing THNFs with a minimum amount of catalysts and evaluate the recyclability compared to the suspended THNFs throughout repeated cycles of photocatalytic degradation of BPA under the optimum operating parameters

1.4 Scope of Study

To achieve those objectives mentioned in the previous sections, the scopes of this study is outlined as follows:

1. Synthesizing THNFs via template synthesis using electrospun PAN nanofiber as the template and calcining at different temperatures (400, 500, and 600 °C) with constant duration and heating rate (4h, 2 °C/min)

2. Characterizing the morphology and evaluating the photocatalytic properties of the resultant TiO_2/PAN-NF and THNFs using field emission scanning electron microscopy (FESEM), energy dispersive x-ray (EDX), Fourier-transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), X-ray diffraction (XRD), nitrogen adsorption-desorption and UV-Vis spectrophotometer. The photocatalytic degradation for 10 ppm of BPA was conducted under UV light and the BPA concentration was measured using high-performance liquid chromatography (HPLC).

3. Measuring the photocatalytic performance of THNF600 in degradation of BPA by varying the THNFs dosage (0.25, 0.50, 0.75, and 1.0 g/L). The pH and initial BPA concentration were constant at pH 7.4 and 10 ppm respectively.

4. Measuring the photocatalytic performance of THNF600 in degradation of BPA with different initial pH of the solution (4.1, 7.4, and 11.3). The dosage of TiO_2 and initial BPA concentration were kept constant at 0.50 g/L and 10 ppm, respectively.

5. Measuring the photocatalytic performance of THNF600 in degradation of BPA with a different initial concentration of BPA (10, 20, 30, 40, and 50 ppm). The dosage of TiO_2 and pH were kept constant at 0.50 g/L and pH 7.4, respectively.

6. Experimenting with BPA photodegradation using THNF600 and FS-THNFs throughout five cycles under optimum operating parameters. The recyclability of THNF was evaluated by repeating the degradation of BPA under optimum conditions (THNF dosage = 0.75 g/L, pH = 4.1, BPA concentration = 10 ppm).

1.4 Significance of Study

Titanium dioxide is a suitable candidate as the photocatalyst material because of its high hydrophilicity, chemical stability, long durability, nontoxicity, low cost, and transparency to UV light. The utilization of TiO_2 in the form of hollow nanofibers enhances photocatalyst performance due to extensive specific surface areas, larger accessible active sites, and higher aspect ratios. Due to the calcination process that results in powdered form catalyst, the TiO_2 hollow nanofibers photocatalysts need to undergo a difficult catalyst recovery process to be reused in the next degradation cycles. The integration of synthesized THNFs into a free-standing form is advantageous because it eliminates the post-recovery process of small-sized catalysts and reduces the operating time and cost. Thus, the application of free-standing THNFs will lead to efficient filtration and photocatalytic degradation of organic pollutant.

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LIST OF PUBLICATIONS

Journal with Impact Factor

1. **Mohammad Jafri, N. N.**, Jaafar, J., Alias, N. H., Samitsu, S., Aziz, F., Wan Salleh, W. N., et al. (2021). Synthesis and Characterization of Titanium Dioxide Hollow Nanofiber for Photocatalytic Degradation of Methylene Blue Dye. *Membranes*, 11(8), 581. https://doi.org/10.3390/membranes11080581 **(Q1, IF: 4.106)**

Indexed Conference Proceedings

1. **Jafri, N. N. M.**, Jaafar, J., Othman, M. H. D., Rahman, M. A., Aziz, F., Yusof, N., et al. (2021). Titanium dioxide hollow nanofibers for enhanced photocatalytic activities. In *Materials Today: Proceedings.* (pp. 2004-2011) https://doi.org/10.1016/j.matpr.2021.02.662 **(INDEXED by SCOPUS)**