

NANOCOMPOSITE OF BISMUTH FERRITE AND ACTIVATED CARBON FOR
PHOTOCATALYTIC DISINFECTION OF MICROBE

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DEDICATION

This thesis is dedicated to my beloved mom and dad, Zaiton binti Suhut and Daub bin Hashim who taught me that the best kind of knowledge to have is that which is learned for its own sake. I also dedicated this thesis to my siblings who always give me an endless encouragement. Last but not least, this thesis is dedicated to my supportive friends, Eiza Fareeza and Nurul Syazwani for always offering me an emotional support and genuine reassurance. May this thesis be an inspiration and guidance in future.

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ABSTRACT

Microbial pathogenic contaminants such as bacteria, viruses and protozoa pose a major threat to environment human health that can cause deadly infectious diseases. One promising way of disinfection activity is by photocatalysis. Bismuth ferrite (BFO) has been regarded as an efficient visible-light driven material for photocatalyst due to its narrow band gap value. However, rapid recombination of photogenerated electron (e^-) – hole (h^+) pairs has limits its application as photocatalyst. To overcome this, BFO-activated carbon nanocomposites (BFO-AC) was synthesized by ultrasonication method with various ratio of activated carbon. Characterization using X-Ray diffraction analysis showed no change in crystallinity of BFO nanoparticles when activated carbon was incorporated into nanoparticles. By using the UV-Vis diffuse reflectance spectroscopy (UVDRS), the emission band of all BFO and BFO-AC nanocomposites were found within visible light range (400-700 nm) and BA (1:1.5) was having the lowest band gap value of 1.86 eV. Interestingly, after the addition of AC, the Brunauer–Emmett–Teller (BET) surface area of BA (1:0.5), BA (1:1) and BA (1:1.5) dramatically increased ie., 267.51 m²/g, 351.82 m²/g and 862.99 m²/g, respectively. BET results indicate BA (1:1.5) has the highest surface area due to its porous property. The field emission scanning electron micrograph has shown that BA (1:1.5) possess a better distribution and less agglomeration. The photoluminescence analysis demonstrated the intensity of all BFO-AC nanocomposites decreases compared to pristine BFO. The decrease of photoluminescence indicate the lower rate of electron (e^-) – hole (h^+) pairs recombination. Photocatalytic disinfection of *S.aureus* by AC, BFO, BA (1:0.5) and BA (1:1) were obtained within 150 min, 120 min, 120 min and 90 min, respectively. BA (1:1.5) exhibited the strongest bactericidal activity as a complete inactivation of *S.aureus* was achieved within 60 min. The surface and morphology of *S.aureus* were characterized by transmission electron microscopy analysis. Bacterial cell had a smooth and spherical shape before being irradiated under visible light. However, the bacterial cell were severely damaged and ruptured as the irradiation time increased, implying that *S.aureus* was killed. It is herein worth noting that the incorporation of AC onto BFO significantly improved the performance of photocatalytic disinfection of *S.aureus* under visible-light irradiation.

ABSTRAK

Bahan cemar patogen mikroba seperti bakteria, virus dan protozoa menimbulkan ancaman besar terhadap isu alam sekitar serta mengancam kesihatan manusia kerana ia boleh menyebabkan penyakit yang membawa maut. Salah satu cara yang boleh menjanjikan pembasmian kuman adalah dengan fotomangkin. Bismuth ferrite (BFO) telah dianggap sebagai bahan fotomangkin di pacu cahaya tampak yang berkesan kerana mempunyai nilai jalurnya yang kecil. Walaubagaimanapun, penggabungan semula pasangan lubang elektron yang pantas menghadkan penggunaannya sebagai fotomangkin. Untuk menangani permasalahan ini, nanokomposit BFO-karbon teraktif (BFO-AC) telah disintesis melalui kaedah ultrasonik dengan nilai nisbah karbon teraktif yang berbeza. Pencirian menggunakan analisis pembelauan sinar-X menunjukkan tiada perubahan dalam kehabluran nanopartikel BFO apabila karbon teraktif dimasukkan ke dalam nanopartikel. Dengan menggunakan spektroskopi pemantulan resap UV-Vis (UVDRS) jalur pancaran semua nanokomposit BFO-AC dan BFO berada dalam julat cahaya nampak (400-700 nm) dan BA (1:1.5) mempunyai nilai jurang jalur paling rendah iaitu 1.86 eV. Menariknya, selepas penambahan AC, luas permukaan Brunauer-Emmett-Teller (BET) BA (1:0.5), BA (1:1) dan BA (1:1.5) masing-masing meningkat setiap satunya adalah 267.51 m²/g, 351.82 m²/g dan 862.99 m²/g. Analisis BET menunjukkan BA (1:1.5) mempunyai luas permukaan paling tinggi disebabkan oleh sifat berliang. Analisis mikroskopik elektron pengimbasan pelepasan medan menunjukkan bahawa BA (1:1.5) mempunyai taburan yang lebih baik dan kurang penggumpalan. Analisis foto pendarcahaya menunjukkan keamatan bagi semua BFO-AC nanokomposit berkurangan berbanding dengan BFO. Pengurangan foto pendarcahaya menunjukkan penggabungan pasangan lubang elektron dengan kadar yang lebih rendah. Pembasmian kuman fotopemangkin *S.aureus* oleh AC, BFO, BA (1:0.5) dan BA (1:1) masing-masing diperoleh dalam masa 150 min, 120 min, 120 min dan 90 min. BA (1:1.5) menunjukkan penyahaktifan bakteria yang paling tinggi dalam masa 60 min. Permukaan dan morfologi *S.aureus* dicirikan melalui analisis mikroskopi elektron transmisi. Sel bakteria berbentuk sfera dan mempunyai permukaan yang licin sebelum disinari dibawah cahaya tampak. Walaubagaimanapun, sel bakteria pecah dan rosak apabila masa sinaran meningkat dan ini membuktikan bahawa *S.aureus* telah dimatikan. Secara ringkas, penggabungan AC kepada BFO telah menunjukkan peningkatan prestasi pembasmian fotopemangkin kuman *S.aureus* di bawah penyinaran cahaya tampak.

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LIST OF ABBREVIATIONS

AOPs	-	Advanced Oxidation Process
BET	-	Brunauer–Emmett–Teller
BFO	-	Bismuth Ferrite
BFO-AC	-	Bismuth Ferrite-Activated Carbon
BJH	-	Barrett–Joyner–Halenda
DI	-	Deionized Water
FESEM	-	Fourier Electron-Scanning Electron Microscope
FTIR	-	Fourier Transform Infrared
IUPAC	-	International Union of Pure and Applied Chemistry
PL	-	Photoluminescence
ROS	-	Reactive Oxygen Species
TiO ₂	-	Titanium Dioxide
TEM	-	Transmission Electron Microscopy
TOC	-	Total Organic Carbon
UV	-	Ultraviolet
UVDRS	-	Ultraviolet-Diffuse Reflection Spectra
WHO	-	World Health Organization
XRD	-	X-ray powder diffraction

LIST OF SYMBOLS

CFUL/mL	-	Colony Forming Unit/ millilitre
h	-	Hour
Mg	-	Miligram
L	-	Litre
°C	-	Degree Celcius
µm	-	Nanometre
g	-	Gram
%	-	Percentage
M	-	Molarity
Min	-	Minutes

CHAPTER 1

INTRODUCTION

1.1 Research Background

Water is the most essential and fundamental to human population across the world. Human beings need water for drinking, cooking, washing, irrigated agricultural and other socioeconomic activities. Only about 1% of water is consumable for human consumption (Anjum *et al.*, 2019). With the increasing population growth rate, the world's population is expected to reach over 9 billion people by the year 2050. Microbial contamination includes bacteria, viruses, and parasites in water has become significantly cause a great threat to human and other lives in developed and developing countries. According to the World Health Organization (WHO), severe lack of cleanliness and inadequate knowledge of sanitation cause 80% of disease in humans across the world (Punia *et al.*, 2021). Concerns related to water safety plays an important role in sustaining life. Contaminated water sources contain pathogenic microorganisms which lead to various of serious diseases such as diarrhea, cholera, typhoid, polio and dysentery (Pandey *et al.*, 2014). More than 3.5 million people die each year due to waterborne diseases. The treatment of contaminated water can reduce these concerns to provide safe and readily available water (Pinki *et al.*, 2021).

Different methods have subsequently been investigated to improve the efficiency of treating microbially contaminated water. Despite many conventional water disinfection techniques such as chlorination, ozonation, UV irradiation, these application are still limited to the removal of microorganisms. In a way to efficiently remove microbial, this research has been focus on photocatalysis, a green solution technology that possess a vital role for antimicrobial disinfection application by generating reactive oxygen species (ROS) which are responsible for damaging the cell components of bacteria in the presence of light (Ganguly *et al.*, 2018).

The discovery of water splitting experiment was reported by Fujishima and Honda, (1972) by using titanium dioxide (TiO_2) photocatalyst in the presence of solar light. This pioneered research led to developing numerous photocatalyst for degradation of various contaminants. Further, Matsunaga *et al.* (1985) studied the photocatalytic disinfection of *Escherichia coli* (*E.coli*), *Saccharomyces Cerevisiae* and *Lactobacillus Acidophilus* using TiO_2 under UV light. Following this, a lot of studied have investigated photocatalytic disinfection by various photocatalyst including graphitic carbon nitride- silver bromide (g- C_3N_4 -AgBr), iron-dope bismuth vanadate (Fe-doped BiVO_4), molybdenum disulfide (MoS_2) and tungsten disulfide (WS_2) nanoparticles, carbon supported vanadium tetrasulfide (VS_4/CP), cadmium sulfide (Cds), copper-titanium dioxide (Cu-TiO_2) nanofibers and lead-bismuth ferrite/reduced graphene oxide ($\text{Pb-BiFeO}_3/\text{rGO}$). However, according to Kumar *et al.* (2021), there are still very few study on bismuth based photocatalytic disinfection activity. To harness solar light, bismuth ferrite (BFO) has been regarded as one of the promising photocatalyst due to their low band gap value ($\sim 2.2\text{eV}$) and possessed magnetic properties. Howbeit, the application of BFO alone was practically restricted by rapid recombination of photo-generated electron – hole ($e^- - h^+$) pairs (Li *et al.*, 2019). An attempt needs to be made in order to circumvent this limitation. Thereupon, the modification of BFO needs to be emphasized to enhance photocatalytic performance of visible light-responsive photocatalysts.

Photocatalyst combine with carbonaceous material have been attempted to surmount these shortfalls in photocatalytic performance (Yahya *et al.*, 2018). The application of carbonaceous material such as activated carbons (AC) to support photocatalyst has been widely applied by researchers to improve photocatalytic efficiencies. According to Saravanan *et al.* (2021) nanocomposite photocatalyst which comprise of metals or metal oxides have been showing excellent photocatalytic performance. Activated carbon has a remarkable properties including high adsorption capacity, large surface area, microporous structure, high thermal stability and environmental applications (Alhan *et al.*, 2019). AC greatly promote the separation of photo-induced $e^- - h^+$ pairs and improve the activity of photocatalysis (Chen *et al.*, 2017). Various studies have shown interesting characteristics upon integration of

adsorbent with photocatalyst thus, improving the overall photocatalytic efficiency (Yahya *et al.*, 2019).

1.2 Problem Statement

The most widely studied photocatalyst, TiO₂ are facing crucial problems, wide band gap (3.2-3.35 eV) that limits the utilization of visible-light and resulted in low quantum efficiency (Gamage and Zhang, 2010). Thus, BFO is the potential candidate as the photocatalyst due to their intrinsic properties such as low band gap (~2.5 eV) compared to commercial TiO₂. Moreover, it also has an outstanding properties as a photocatalyst due to its non-toxic nature, excellent chemical stability, low cost, magnetic properties, efficient response to visible light irradiation and have been proved to have a good antimicrobial properties. This photocatalyst shows a comprehensive properties compare to other materials. Despite these promissory findings, there are some limitation of BFO that needs an improvement in which designing of more efficient nanostructures including fast recombination of photogenerated ($e^- - h^+$) pairs. Separation of photogenerated ($e^- - h^+$) pairs subsequently influence the performance of photocatalytic activity (Wei *et al.*, 2017). In order to further improve the practical applications of BFO photocatalyst in photocatalytic activity, the modification on BFO needs to be emphasized to enhance the properties of BFO.

The synergistic effects between AC and semiconductor significantly influence the photocatalytic activity (Li *et al.*, 2019). Incorporation of AC remarkably prevent the ($e^- - h^+$) pairs recombination in photocatalytic activity (Ramya *et al.*, 2018). AC have an outstanding potential as adsorbent due to its fascinating properties such as microporous structure, large surface area and high adsorption capacity (Yahya *et al.*, 2018). Moreover, due to its good biocompatibility, AC can remove microorganisms which can play a part in water treatment (Devi, Mohanta and Ahmaruzzaman, 2019). Yamamoto, Sawai and Sasamoto (2002) also reported AC has an excellent affinity to microorganisms and adsorbed large amount of bacteria. These intriguing properties of AC increase its use as a stable supporting material for the synthesis of BFO.

An efficient bacterial inactivation often entails the interruption or complete destruction of their physiological functions including cell membrane (Zhang *et al.*, 2018). The cell membrane appears to be an important first target by ROS species in disinfection activity leading to the loss of membrane permeability. The destruction of bacterial cell membrane during photocatalytic disinfection subsequently result in the releasing of intracellular components (Zhang *et al.*, 2019). Hence, the changes of microbial morphology to further validate the efficiency of bacterial inactivation.

1.3 Research Objectives

The aim of this study is to synthesis BFO: AC nanocomposites for adsorption and photocatalytic microbial disinfection present in water under visible-light irradiation. In order to achieve that, there are three specific objectives in this study, which are:

- i. To assess the effects of activated carbon (AC) ratio incorporated onto BFO via ultrasonication method on physicochemical properties of the synthesized BFO-AC nanocomposites.
- ii. To identify the effects of irradiation time and BFO-AC ratio on antimicrobial activity of BFO-AC nanocomposites on Gram-positive bacteria (*Staphylococcus aureus*).
- iii. To establish the disinfection mechanism on a subcellular level via systematic analysis focusing on cell morphology.

1.4 Scope of Study

- i. Synthesizing pure BFO using sol-gel auto combustion method with calcination temperature at 500°C for 3 h.
- ii. Synthesizing BFO-activated carbon (BFO-AC) with different activated carbon ratio using ultrasonication method for 3 h.

- iii. Characterization of physicochemical properties of the synthesized pure BFO and BFO: AC nanocomposites by using several characterizations such as X-ray diffractometer (XRD), UV-Vis diffuse reflectance spectroscopy (UVDRS) analysis, Brunauer-Emmet-Teller (BET), Fourier Transform Infrared Spectroscopy (FTIR), field emission scanning electron microscopy (FESEM) and photoluminescence analysis (PL).
- iv. Performing photocatalytic disinfection study of *S.aureus* with 1 mg catalyst loadings at pH 7 under visible light irradiation. Initial bacterial concentration is 3×10^6 CFU/ml.
- v. Evaluation of bacterial cell death by Transmission Electron Microscopy (TEM) analysis.

1.5 Significance of Study

Photocatalysis, is an effective method to oxidize many organic contaminants at ambient conditions. Thus, researches on synthesizing visible-light driven photocatalyst that are easy to be produced and large scalable nanoparticles with desired properties are the main priorities. In this study, the synthesized BFO photocatalyst was prepared via sol-gel auto combustion and activated carbon was incorporated onto BFO via ultrasonication method. Activated carbon was considered as better adsorbents due to its large surface area, microporous structure as well as reduce the recombination rate of charge carriers ($e^- - h^+$) pairs. Additionally, varying different weight ratios of activated carbon onto BFO was proved to significantly enhance the efficiency of photocatalytic activity. In fact, this study shows the bacterial cell wall was severely damaged upon irradiation of visible light at different time intervals. The synthesized BFO-AC nanocomposites led to significant improvement in photocatalytic disinfection of *S.aureus* under visible light irradiation. The utilization of activated carbon supported semiconductor provides as an effective alternative in photocatalytic disinfection activity.

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LIST OF PUBLICATIONS

Article paper

1. **Nur Atiqah Daub**, Farhana Aziz, Madzlan Aziz, Juhana Jaafar, Wan Norhayati Wan Salleh, Norhaniza Yusof & Ahmad Fauzi Ismail (2020). A Mini Review on Parameters Affecting the Semiconducting Oxide Photocatalytic Microbial Disinfection. *Water, Air & Soil Pollution*. 231 (461) <https://doi.org/10.1007/s11270-020-04829-y>

Conference proceeding

1. **Nur Atiqah Daub**, Farhana Aziz, Nor Azimah Mohd Zain, Lau Woei Jye, Norhaniza Yusof, Wan Norhayati Wan Salleh & Juhana Jaafar (2020). Photocatalytic disinfection under visible light irradiation by BFO photocatalyst. *IOP Conf. Series: Materials Science and Engineering*. 1142 (1) DOI:10.1088/1757-899X/1142/1/012002.
2. **Nur Atiqah Daub**, Farhana Aziz, Lau Woei Jye & Nor Azimah Mohd Zain (2022). Synthesis of Bismuth Ferrite-Activated Carbon (BFO-AC) Nanoparticle and Their Characterization. *AIP Conference Proceedings*.

Book chapter

1. **Nur Atiqah Daub**, Farhana Aziz, Arif Aizat, Norsyazwani Yahya (2020). Synthetic Polymer-based Membranes for Photodegradation of Organic Hazaedous Materials. *Synthetic Polymeric Membranes for Advanced Water Treatment, Gas Separation, and Energy Sustainability*. 53 (70) 10.1016/B978-0-12-818485-1.00004-6