PHOTOCATALYST COMPOSITES OF ZINC OXIDE-DOPED TITANIUM DIOXIDE MODIFIED WITH SODIUM SILICATE FOR ANTIBACTERIAL APPLICATIONS

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ABSTRACT

Superbugs have troubled and challenged mankind since the development of antibiotics could not keep up with the rate of bacterial evolution. To avoid cross-contamination, the emphasis should be placed on effective protection beginning with the surroundings. This study concentrated on the rapid sonochemical synthesis of photocatalyst sodium silicate loaded titanium dioxide and zinc oxide (TiO_2@ZnO_Na_2SiO_3) composites as an antibacterial agent using short synthesis time and less hazardous solvents. Anatase was obtained as evidenced by X-ray diffraction (XRD) and high resolution transmission electron microscopy (HRTEM) analyses. Meanwhile, bonding present between the main element of the synthesized sample were confirmed by Fourier transform infrared spectroscopy (FTIR). Visible range was obtained for TiO_2@ZnO samples while near ultraviolet range was obtained for TiO_2@ZnO_Na_2SiO_3 samples as depicted by diffused reflectance ultraviolet-visible spectroscopy (DR UV-Vis). All TiO_2@ZnO have lower recombination rate compared to ZnO whereas all TiO_2@ZnO_Na_2SiO_3 samples have lower recombination rate compared to TiO_2@ZnO (TiO_2:ZnO = 1:0.1) composite as observed under fluorescence spectroscopy. All composites have irregular shape as noticed under scanning electron microscopy (SEM). Variation of ratios in TiO_2:xZnO (x = 0.1, 0.2, 0.3, 0.4, and 0.5) and TiO_2@ZnO_yNa_2SiO_3 volume percent (y = 2, 4, 6, 8, and 10) were made to obtain the best ratio for highest antibacterial activity. TiO_2@0.1ZnO was determined to be the optimal ratio by evaluating both physiochemical properties and antibacterial performance. TiO_2@0.1ZnO had a bacteria killing efficiency (BKE) of 78.68% against *S. aureus* and 99.99% against *E. coli*, due to its smallest crystallite size (55 nm), lowest band gap energy (2.68 eV), and lower recombination rate. The optimal ratio of TiO_2@ZnO_10Na_2SiO_3 was obtained after further modification of previous ratio of TiO_2@0.1ZnO with Na_2SiO_3, which achieved a lower band gap energy (3.10 eV) and the lowest rate of recombination amongst other variants, with a BKE of 81.36% against *S. aureus* and 99.99% against *E. coli*. As seen under HRTEM image and amorphous XRD pattern, the loading of Na_2SiO_3 outside of TiO_2@ZnO was successful. Therefore, this study has successfully synthesised a new antibacterial agent TiO_2@ZnO_Na_2SiO_3.

ABSTRAK

‘*Superbug*’ telah mengganggu dan mencabar manusia disebabkan penemuan antibiotik tidak dapat mengikuti kadar evolusi bakteria. Tumpuan harus diletakkan pada perlindungan yang betul bermula dengan alam sekitar untuk mengelakkan pencemaran silang. Kajian ini tertumpu kepada sintesis sonokimia pesat fotomangkin TiO_2@ZnO_Na_2SiO_3 sebagai agen antibakteria dengan menggunakan masa sintesis yang singkat dan pelarut yang kurang berbahaya. Anatase diperolehi seperti yang ditunjukkan oleh analisis pembelauan sinar-X (XRD) dan mikroskopi penghantaran elektron beresolusi tinggi (HRTEM). Kehadiran ikatan di antara TiO_2@ZnO_Na_2SiO_3 telah disahkan oleh spektroskopi infra merah transformasi Fourier (FTIR). Selain itu, panjang gelombang berhampiran cahaya nampak telah didapati untuk TiO_2@ZnO_Na_2SiO_3 melalui spektroskopi pantulan terbaur ultralembayung-nampak (DR UV-Vis). Semua TiO_2@ZnO mempunyai kadar rekombinasi yang rendah berbanding ZnO manakala semua TiO_2@ZnO_Na_2SiO_3 mempunyai kadar rekombinasi yang lebih rendah berbanding TiO_2@ZnO (TiO_2: ZnO = 1: 0.1) seperti yang diperhatikan di bawah spektroskopi pendarfluor. Semua komposit mempunyai bentuk yang tidak sekata seperti yang diperhatikan dalam mikroskopi imbasan elektron (SEM). Variasi dalam nisbah komposit TiO_2@xZnO (x = 0.1, 0.2, 0.3, 0.4, dan 0.5) dan TiO_2@ZnO_yNa_2SiO_3 peratus isipadu (y = 2, 4, 6, 8, dan 10) telah dibuat untuk mengoptimumkan bahan ini. TiO_2@0.1ZnO telah ditentukan sebagai nisbah optimum dengan menilai kedua-dua sifat fisiokimia dan prestasi antibakteria. Nisbah ini mempunyai kecekapan membunuh bakteria (BKE) sebanyak 78.68% terhadap *S. aureus* dan 99.99% terhadap *E. coli*, serta sifat fizikalnya seperti saiz kristal yang terkecil (55 nm), tenaga jurang jalur yang terendah (2.68 eV) dan kadar penggabungan semula yang lebih rendah. Nisbah optimum TiO_2@ZnO_10Na_2SiO_3 diperolehi selepas pengubahsuaian dengan nisbah komposit TiO_2@0.1ZnO. Ia mencapai tenaga jurang jalur yang lebih rendah (3.10 eV), kadar penggabungan semula yang paling rendah di antara varian lain dan BKE 81.36% terhadap *S. aureus* dan 99.99% terhadap *E. coli*. Seperti yang ditunjuk oleh HRTEM dan XRD, penempatan Na_2SiO_3 di bahagian luar TiO_2@ZnO adalah berjaya. Oleh itu, kajian ini telah berjaya mensintesis agen antibakteria TiO_2@ZnO_Na_2SiO_3 yang baharu.

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LIST OF ABBREVIATIONS

AFM	Atomic Force Microscopy
Ag	Silver
ATP	Adenosine 5'-triphosphate
$CaCO_3$	Calcite
cm	Centimetre
cm^2	Square centimetre
cm^{-1}	Per centimetre
DNA	Deoxyribonucleic acid
DR UV Vis	Diffuse Reflectance Ultraviolet-Visible Spectroscopy
EDX	Energy Dispersive X-ray
FESEM	Field Emission Scanning Electron Microscopy
FTIR	Fourier Transform Infrared Spectroscopy
HRTEM	High Resolution Transmission Electron Microscopy
H_2O	Hydrogen oxide/water
H_2O_2	Hydrogen peroxide
$HO_2\bullet$	Hydroperoxyl radical
IR	Infrared
ISO	International Organization for Standardization
JCPDS	International Centre for Diffraction Data
JIS Z	Japanese Industrial Standard Miscellaneous
M	Molar
MB	Methylene blue
mL	Millilitre
NaOH	Sodium hydroxide

Na_2SiO_3	Sodium silicate
nm	Nanometre
$\bullet O_2^-$	Superoxide radical
$\bullet OC_4H_9$	t-butoxy radical
•OH	Hydroxyl radical
PVC	Pigment volume concentration
ROS	Reactive oxygen species
TEM	Transition Electron Microscopy
TiO_2	Titanium dioxide
$Ti(OH)_4$	Titanium hydroxide
TBOT	Titanium butoxide
UV	Ultraviolet
μL	Microlitre
μm	Micrometre
Vis	Visible
wt	Weight
XPS	X-ray Photoelectron Spectroscopy
XRD	X-ray Diffraction
ZnO	Zinc Oxide

LIST OF SYMBOLS

Å	Angstrom
e^-	Electron
eV	Electron volt
h	Planck constant
hν	Energy of photon
h^+	Hole
Hz	Hertz
W	Watt
2θ	2 Theta
°	Degree
℃	Degree Celsius
λ	Lambda
%	Percent
Ω	Ohm

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CHAPTER 1

INTRODUCTION

1.1 Research Background

People were made aware of the existence of the virus by the epidemic of coronavirus disease, or COVID-19, which began in early 2020. Based on a survey poll done with citizens from 11 countries, the majority of them believe that protect themselves with good hygiene could prevent themselves from infected (Company, 2020). In fact, dangerous microorganisms including bacteria, fungus, protozoa, and helminths are all around us in addition to viruses. (Madeline, 2010). Hospital-acquired infections caused by gram-positive and gram-negative bacteria had been infected more than 2 million people and contribute to 23,000 deaths or even more in developing countries. This also caused huge economic losses in terms of medicines, therapies, and maintaining a safe environment in the hospital that propitious to the growth of microorganisms (Bianca *et al.*, 2022). Microbes tend to form biofilm on surface and they are 1000-fold more resistance towards antibiotics which poses a challenge on medical field (Hans-Curt *et al.*, 2019). Some bacteria could survive at room temperature such as *Mycobacterium tuberculosis* and *Escherichia coli* for hours to weeks while some could even withstand extreme temperature and humidity like *Staphylococcus aureus* (Fazilet *et al.*, 2019). Other than vaccines and medicines, some precautionary habits have been developed or educated to prevent or treat infectious diseases.

There are few existing antibiotics target certain bacteria but overuse of the antibiotics has caused the emergence of ‘superbugs’, multi-drug resistance microorganisms. As a result, at least 700,000 deaths per year and was projected to rise to 10 million by the year 2050. Concerns arise when the creation of antibiotics does not reach the speed of microorganism mutation, especially in the hospital where vulnerable patients were in their recovering stages and could be danger by these superbugs. New antibacterial materials that provide long-lasting, resilience and are effective in antibacterial activities are gaining interest (McCulloch *et al.*, 2022; Zhe *et al.*, 2022). Some cleaning products that contain alcohol, bleach or ammonium compounds could reduce almost 99.9% of the bacteria on household surfaces through disinfection however care should be taken when using these products to maximize their effect. Disinfectants or organic biocides usually provide short-term effects, harmful to human and the environment, and also, potentially build an environment that bacteria resist due to mutagenesis. Inorganic materials were found to be a more effective approach for antimicrobial (Maya *et al.*, 2018; Zhe *et al.*, 2022). The morphological and physicochemical properties of inorganic particles such as close interaction with microbial membranes had supported it to be an effective antimicrobial applications (Emmanuel *et al.*, 2020).

The protective coating, paint was innovated in ancient times as cave painting which is now the coating on walls of hospitals, commercial buildings, or houses. It is composed of pigments, resins, solvents, and additives that contribute to its color, adhesion to walls and functionalities (Khanna, 2015; Pilotek *et al.*, 2005). The existence of microbes not only harms human but also leads to biodeterioration of building materials through their complex metabolic activities. For instance, organic acid produced by *Fusarium oxysporum* could deteriorate concrete and weaken its lifespan (Farooq *et al.*, 2015). Of late, the development of antibacterial paint started to add organic or inorganic biocides as an additive in paint to reduce or control the effect caused by microbes including bacteria, fungi, and viruses. Organic biocides such as alcohols or quaternary ammonium do not last long and could be the nutrient source of microbes that attributes to their growth and developed resistance (Mian *et al.*, 2019). Modification of physical properties including surface wettability and morphology had been done to discover a better photocatalyst as the additive in antimicrobial paint (Gibyoung *et al.*, 2017; Sudipto Pal *et al.*, 2016; Suélen *et al.*, 2019).

An example of inorganic biocides, titanium dioxide or titania (TiO_2) is normally known to be non-toxic, highly durable with a high refractive index and low cost (Mantravadi, 2017; Prashant, 2002). In industries, TiO_2 has been commonly used as a pigment in paint manufacturing, production of sunscreen, toothpaste, and coloring agent (Philippe, 2018). Also, several metals and metal oxides such as Ag, Au, SiO_2, and ZnO have been doped into/onto TiO_2 in order to improve the photocatalytic efficiency of the materials (Trilok *et al.*, 2019; Verma *et al.*, 2020). Zinc oxide (ZnO) is easy to be synthesized, inexpensive, and is known to have a band gap energy of 3.37 eV and is approved to use under recommended amount by Food and Drug Administration (Al-Tayyar *et al.*, 2020). Rubber, paint, coating, concrete production, electronics, and cosmetics are examples of its industrial applications due to their diverse function from durability, antimicrobial, ultraviolet, and visible light resistance (Jinhuan *et al.*, 2018). Previous studies documented that ZnO portrayed low toxicity and a good antimicrobial property due to hydrogen peroxide generated and cell membrane destruction by Zn^{2+} (Mastan *et al.*, 2020). Similar to TiO_2, ZnO also could perform antibacterial activities under light irradiation which its product reactive oxygen species (ROS) could not only attack bacteria but also the organic components in paint such as organic binder (Arekhi *et al.*, 2018).

Silicate binders like sodium silicate (Na_2SiO_3) was proven to be a good replacement for the organic binder that was able to cure by the evaporation of water (Gettwert *et al.*, 1998; Parashar *et al.*, 2003). It served to reduce the effects of photocatalysts on all organic components due to antibacterial activities that deteriorated the essential elements in paint. Sodium silicate is cheap, non-toxic, available, and has been used to support metal nanoparticles in many coating applications such as paint and face masks (Askwar *et al.*, 2012; Bassi *et al.*, 2018; Parashar *et al.*, 2003; Sri Bala Jeya *et al.*, 2022). It has stupendous advantages such as promoting photoactivities and pollutants absorption, however, the optimum dosage should be controlled to avoid some disadvantages such as agglomeration which retarded the photoactivities (Arekhi *et al.*, 2018; Petržílková, 2019).

Numerous synthesis methods such as sol-gel, hydrothermal, and pulsed laser ablation have been used to synthesize the photocatalyst. These synthesis methods, however, give certain undesired disadvantages such as the usage of toxic solvent, long synthesis duration, and small product yields. Thereby, sonochemical synthesis could be served as a promising approach to synthesizing materials with high homogeneity, phase purity, and small size (Chitra *et al.*, 2018).

Due to the potential of both TiO_2 and ZnO as photocatalysts and Na_2SiO_3 as the inorganic binder that makes them suitable as the antibacterial additive for paint. Therefore, this research aimed to synthesize a novel photocatalyst that involved the composites of TiO_2 and ZnO (TiO_2@ZnO) with the synergy of inorganic binder, Na_2SiO_3 as a durable antibacterial additive in paint. Although TiO_2@ZnO photocatalysts have been widely developed, based on current knowledge, there are less research has combined inorganic binder Na_2SiO_3 in the photocatalyst composite of TiO_2 and ZnO in paint application yet. On top of that, the efficient sonochemical synthesis method could help to synthesize the photocatalyst composite in shorter duration and much more environmental-friendlier experimental conditions. An attempt was made in this work to synthesize TiO_2@ZnO_Na_2SiO_3 via the sonochemical method.

1.2 Problem Statement

Superbugs had alarmed people to create new resilience materials to deal with them. Despite the presence of antibiotics and disinfectants, inorganic materials are the potential to tackle this issue and thus produce a safe environment with safe surfaces. TiO_2 and ZnO are examples of inorganic photocatalysts which have been proven for their efficient photocatalytic activity. Under light irradiation, the photogenerated electron hole pairs could generate reactive oxygen species (ROS) to kill bacteria.

However, the broad bandgap of both TiO_2 and ZnO at 3.2 eV only allows them to be active in the ultraviolet region. Moreover, both photocatalysts suffer from high recombination rates and short lifetime of charge carriers if without any modifications. It was suggested that the combination of them could reduce the recombination rate by improving the charge separation efficiency. The synergy of TiO_2 and ZnO could also improve the performance of the composites under visible light. The durability of paint here could be also understood as the paint's appearance long-lastingness. Short lifespan was observed when antibacterial paint did not optimize their poor durability caused by the deterioration of organic compounds such as binder and additives in paint that resulted from photocatalysis.

It was claimed previously that the combination of TiO_2-ZnO provided enhancement on antibacterial performance for coating when the dopant ratio of ZnO increased. However, studies usually focused on antibacterial performance instead of the photodegradation issue of organic binder (Yusliza *et al.*, 2020). Of late, inorganic binders such as lime, organosilane, silica sol-gel and water glass had been researched to provide the solution for the organic binder degradation issues. The presence of Na_2SiO_3 was believed to act as a barrier between TiO_2@ZnO and organic components in paint which thus retards the photodegradation of organic components effectively. To ensure effectiveness, the optimum dopant ratio of ZnO and Na_2SiO_3 was studied in this work to achieve a balance between antibacterial and durability.

Some studies reported the various fabrication of TiO_2@ZnO with different synthesis methods like sol-gel, hydrothermal and laser ablation requires a long synthesis duration, more toxic solvents, and expensive advanced instrument (Kalimuthu *et al.*, 2018; Singh *et al.*, 2020). In contrast, the efficient sonochemical synthesis method requires a remarkably shorter synthesis duration, a lesser toxic solvent used, and promote better material phase purity. Specifically, the fabrication of TiO_2@ZnO modified with Na_2SiO_3 through the sonochemical synthesis method has not been reported. Furthermore, the physicochemical properties of synthesized products and their antibacterial performance against selected gram-positive and gram-negative bacteria were worthy of exploration.

In addition, the current reported antibacterial testing methods such as disk diffusion, agar well diffusion, or minimum inhibitory concentration were designed for antibiotics. These methods could not project the antibacterial performance of photocatalysts well as their antibacterial mechanism. Thus, an antibacterial testing method that evaluated the performance of the designed antibacterial agent through surface contact under light irradiation was suggested in this work.

To top it off, despite different combinations of TiO_2 or ZnO with Na_2SiO_3 have been researched before but the previous studies focused solely on antimicrobial applications of the coating. The combination of TiO_2@ZnO with Na_2SiO_3 was therefore an interesting direction to research further to create an antibacterial additive that was potent to have antibacterial function in paint applications. This work particularly researched the sonochemical synthesis of TiO_2@ZnO with Na_2SiO_3 and its optimum dopant ratios of ZnO and Na_2SiO_3 for best surface contact antibacterial performance which has not been reported before.

1.3 Objectives of Study

The objective of this study were:

i. To synthesize titanium dioxide doped with zinc oxide modified with sodium silicate (TiO_2@ZnO_Na_2SiO_3) through sonochemical method;
ii. To characterize the physical and chemical properties of TiO_2@ZnO_Na_2SiO_3 samples;
iii. To optimize the dopant ratio of ZnO and Na_2SiO_3 in TiO_2@ZnO_Na_2SiO_3;
iv. To evaluate the antibacterial properties of TiO_2@ZnO_Na_2SiO_3.

1.4 Scope of Study

This research focused on synthesis, optimization of synthesis parameters, characterizations and evaluation of antibacterial activities of TiO_2@ZnO_Na_2SiO_3. To achieve the first objective, all the samples were synthesized via sonochemical method. For this purpose, precursor of titanium isopropoxide was mixed with ethanol, acetic acid, propylene glycol and ultrapure water then subjected under sonication for minutes at room temperature. It was modified with purchased ZnO under same pot. Different ratio of ZnO to TiO_2 was used to study their differences in antibacterial efficiency. Sodium silicate was mixed with TiO_2@ZnO synthesized earlier in different amount with propylene glycol as dispersing agent and subjected under sonication for another round of minutes.

Afterwards, the surface morphology and particle size of synthesized samples was examined by using Scanning Electron Microscopy (SEM). The atomic structure of the samples was verified by High Resolution Transmission Electron Microscopy (HRTEM). Meanwhile, crystalline phases and band gap energy of the samples were determined by X-ray Diffraction (XRD) and Diffuse Reflectance Ultra-Violet Spectroscopy (DR UV-VIS), respectively. The functional group present within the samples were examined using Fourier Transform Infrared Spectroscopy (FTIR). Lastly, the recombination rates were defined by Fluorescence spectroscopy and elemental analysis was obtained from Energy Dispersive X-ray Spectroscopy (EDX).

To achieve Objective 3, dopant ratios of ZnO and Na_2SiO_3 were controlled to optimize antibacterial performance of the designed photocatalysts. A series of TiO_2@ZnO composites of Ti:Zn ratios 1:0.1, 1:0.2, 1:0.3, 1:0.4, 1:0.5 was prepared. The optimum TiO_2-ZnO was chosen based on best physiochemical properties and antibacterial performance. Then, Na_2SiO_3 was added into the best TiO_2@ZnO to form TiO_2@ZnO:Na_2SiO_3 (2v/v%, 4v/v%, 6v/v%, 8v/v%, 10v/v% Na_2SiO_3) under same sonochemical synthesis method as aforementioned.

The antibacterial performance of the synthesized composites was tested according to surface contact antibacterial test under visible light irradiation for 4 hours against gram positive bacteria *Stapylococcus aureus* and gram negative bacteria *Escherichia coli* under visible light irration. The bacteria killing efficiencies (BKE) of the samples were calculated.

1.5 Significance of Study

The invasion of bacteria and superbugs has brought the concern to people and hence innovations are needed urgently to prevent and protect their life. Otherwise, infectious diseases will disrupt of daily activities, then a raising in the death rate which in turn affects the economy of a nation. Paint as a coating is potential to be researched more on its antimicrobial properties so that it could hinder the growth of bacteria on it which might turn it into a ‘home’ for microorganisms. Cross-contamination might occur when humans get in touch with the microbial on the wall and then to other areas which humans contacts often. Therefore, it is always better to reduce the chances of getting an infection instead of relying on antibiotics which might not be effective after the mutagenesis of microbes. If microbes are not removed from the surface, they might deteriorate the concrete and attributes to the weakening of coating durability.

As a pigment within the paint, titanium dioxide and zinc oxide are well-known for their potential as a photocatalyst and effective antimicrobial properties whereas the combination of these photocatalysts not only could kill bacteria but also slowed the recombination rate through the formation of nanoheterojunction which boost up the photocatalytic efficiency. Inorganic binder, sodium silicate had opened up new opportunities as an alternative for organic resin binder which then makes a more durable paint that resists the attacks from reactive oxygen species which subsequently deteriorate the paint, causing a shorter lifespan.

An environmental-friendlier synthesis method such as the sonochemical method that uses less toxic solvent and gives good phase purity under low temperatures could be explored for novel photocatalyst design. Efficient and effective sonochemical method helps to synthesize photocatalysts in a shorter time frame and could be applied to mix and disperse paint components, forming paint coatings with different properties. Hence, this study could explore the invention of antibacterial additives with efficiency and durability that not only could enhance the fundamental knowledge of efficient sonochemically synthesized material and coating field but also protect the human being and their daily lives by building a safe environment.

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LIST OF PUBLICATIONS

Under the supervision and guidance of my supervisor, Prof Madya Dr Lee Siew Ling, few papers have been successfully submitted or published online as listed below.

1. Chen, M. C., Koh, P. W., Ponnusamy, V. K., & Lee, S. L. (2022). Titanium dioxide and other nanomaterials based antimicrobial additives in functional paints and coatings. *Progress in Organic Coatings*, 163, 106660.
2. Chen, M. C., Koh, P. W., Ponnusamy, V. K., & Lee, S. L. (2022). *Titanium dioxide and other nanomaterials based antimicrobial additives in functional paints and coatings*. International Virtual Symposium in Chemistry: Chemistry Beyond Border 2021, Virtual.