PHOTOCATALYTIC DEGRADATION OF TRICLOCARBAN IN ARTIFICIAL BATHROOM GREYWATER BY MODIFIED TIO_2 NANOTUBES COATED WITH ZEOLITE (TNTs/ZEO)

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DEDICATION

I like to dedicate this achievement to my family especially to my beloved parents, Arifin bin Nor and Ramidah binti Mohd Nor. To my lovely brothers and sisters, Rosliza Arifin, Azmi Arifin, Azman Arifin, Adam Azrin Arifin, Siti Hajarul Ain Arifin, Siti Aisyah Arifin, and my late little brother, Mohammad Zulkifli Razai, thank you for all your supports and always being there for me.

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ABSTRACT

Xenobiotic organic compounds (XOCs) originated from anti-microbial personal care products (PPCPs) including triclocarban (TCC) are emerging contaminants can cause hazardous effect on water environment. The study aims to measure the degradation of the XOCs which is TCC to promote water quality protection measures. Hence, objective of this research is to study the photocatalytic degradation of TCC in greywater. Photocatalytic degradation was enhanced by modification of photocatalyst which is TiO_2 nanotubes with the coating of zeolite (TNTs/Zeo). TNTs/Zeo catalyst were formed by application of electrochemical anodization (ECA) for TiO_2 nanotubes formation and electrophoresis deposition (EPD) in coating zeolite. The characteristics of catalyst were verified using FESEM/EDS, and XRD. In order to control the presence of TCC, the optimization of photocatalytic degradation of TCC was designed via factorize central composite design (FCCD) design matrix of RSM by depending on pH value (3 - 10), TNTs/Zeo catalyst loading size (0.5 cm^2 - 1.0 cm^2) and irradiation time (10 - 60 minutes) as variables. The photocatalytic degradation experiments have been conducted under natural sunlight radiation. The overall finding has directed to the pH value 11, TNTs/Zeo catalyst loading 0.75 cm^2, and irradiation time 50 minutes were the optimum conditions of photocatalytic degradation on TCC that has provided the maximum efficiency of photocatalytic degradation up to 81.2 % removal of TCC concentration. Based on kinetic study by following Langmuir-Hinshelwood (L-H) model and pseudo first order, the significant constant rate obtained at pH 11 which was 0.048 ppm/min, 0.75 cm^2 of TNTs/Zeo catalyst loading size achieved 0.047 ppm/min and 5 ppm of TCC initial concentration reached 0.037 ppm/min. Furthermore, there eleven intermediate products were detected after the whole process of photocatalysis. In addition, the photocatalytic degradation rate of TCC from first and fifth cycles were 94.2 % and 77.4 % where it still can be considered as significant TCC degradation rate.

ABSTRAK

Sebatian organik jenis xenobiotik (XOCs) yang terhasil daripada produk penjagaan diri termasuk triclocarban (TCC) adalah bahan yang boleh mendatangkan kesan sampingan kepada alam sekitar. Dengan itu, objektif kajian ini adalah untuk menyelidik penyingkiran TCC dalam air sisa daripada bilik mandi menggunakan kaedah penyingkiran fotopemangkin. Kaedah ini telah ditingkatkan dengan pengubahsuaian pemangkin iaitu penambahan zeolit pada tiub nano TiO_2 (TNTz/Zeo). Pemangkin TNTs/Zeo dihasil dengan menggunakan kaedah anodisasi elektrokimia (ECA) untuk pembentukan tiub nano TiO_2 serta kaedah pemendapan elektroforesis (EPD) bagi pemendapan zeolit. Ciri-ciri pemangkin TNTs/Zeo telah diklarisifikasi oleh analisis FESEM, EDS dan XRD. Bagi mengawal kewujudan TCC, faktor-faktor bagi proses penyingkiran fotopemangkin terhadap TCC ditentu dengan menggunakan rekabentuk FCCD daripada RSM dan pemilihan faktor-faktor tersebut bergantung kepada pembolehubah iaitu nilai pH (3-10), saiz pemangkin TNTs/Zeo (0.5 cm^2 - 1.0 cm^2) dan tempoh masa proses fotopemangkin (10-60 minit). Eksperimen ini telah dijalankan di bawah penyinaran cahaya matahari. Secara keseluruhan kajian telah membuktikan nilai pH 11, saiz pemangkin 0.75 cm^2 dan masa proses fotopemangkin selama 50 minit adalah ciri-ciri terbaik proses fotopemangkin terhadap TCC di mana kadar penyingkiran kandungan TCC mencapai penyingkiran tertinggi iaitu 81.2 %. Berdasarkan kajian kinetik Langmuir-Hinshelwood (L-H) dan susunan pertama pseudo mendapati kadar malar terbaik penyingkiran TCC adalah pada nilai pH 11 yang mana mencapai kadar malar 0.048 ppm/min, saiz pemangkin 0.75 cm^2 dengan kadar malar 0.047 ppm/min dan 5 ppm kandungan awal TCC telah menjangkau kadar malar 0.037 ppm/min. Selain itu, sebelas produk perantara dikesan selepas satu proses fotopemangkin yang lengkap. Di samping itu, kadar penyingkiran terhadap TCC setelah kitaran pertama dan kelima adalah sebanyak 94.2 dan 77.4 % pengurangan di mana ia boleh diklarifikasikan sebagai kadar penyingkiran TCC yang memuaskan.

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LIST OF SYMBOLS AND ABBREVIATIONS

°C	:	Degree Celsius
μ	:	Micro
*OH	:	Hydroxyl radical
ABGW	:	Artificial bathroom greywater
ANOVA	:	Analysis of variance
AOPs	:	Advance oxidation processes
AP	:	Adequate precision
APHA	:	American Public Health Association
BOD	:	Biochemical oxygen demand
BP3	:	Benzophenone 3
C	:	Carbon
Ca	:	Calcium
CB	:	Conduction band
CdS	:	Cadmium sulfide
Cl	:	Chlorine
cm^2	:	Centimeter square
COD	:	Chemical oxygen demand
DAD	:	Diode array detector
DLLME	:	Disperse liquid liquid microextraction
DO	:	Dissolved oxygen
DOE	:	Department of Environment
DT	:	Deep trap
e^-	:	Electron
EC	:	E. coli
ECA	:	Electrochemical anodization
EDS	:	Energy dispersive spectroscopy
EG	:	Ethylene glycol

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EPD	:	Electrophoresis deposition
EQA	:	Environmental Quality Act
eV	:	Band gap energy
FCCD	:	Factorize central composite design
FDA	:	Food and Drug Association
Fe	:	Iron
g	:	Grams
g/mol	:	Gram per mol
GCMS	:	Gas chromatography microscopy
FKAAB	:	Faculty of Civil Engineering and Built Environment
FESEM	:	Field Emission Scanning Electron Microscope
h^+	:	hole
HCL	:	Hydrochloric acid
HPLC	:	High-performance liquid chromatography
hr	:	hour
hrs	:	hours
K	:	Potassium
k	:	Constant rate
L	:	Liter
L-H	:	Langmuir-Heinshelwood
m	:	meter
mAu	:	Absorbance
min	:	Minutes
Mg	:	Magnesium
mg/L	:	Milligram per liter
n	:	nano
N	:	Nitrogen
Na	:	Sodium
NaOH	:	Sodium hydroxide
NH_4F	:	Ammonium floride
NR	:	Not Recorded
NTU	:	Nephelometric Turbidity
O	:	Oxygen

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PD	:	Photocatalytic degradation
PET	:	Polythylene terephthalate
pH	:	Potential of hydrogen
PPCPs	:	Pharmaceutical and personal care products
ppm	:	Part per million
R^2	:	Correlation coefficient
RSM	:	Response surface methodology
s	:	Second
ST	:	Shallow trap
TCC	:	Triclocarban
TDS	:	Total dissolved oxygen
Ti	:	Titanium
TiO_2	:	Titanium dioxide
TN	:	Total nitrogen
TNTs/Zeo	:	Titanium dioxide nanotubes coated with zeolite
TOC	:	Total organic carbon
TP	:	Total phosphorus
TS	:	Total sulphate
TSS	:	Total suspended solid
UV	:	Ultraviolet
V	:	Voltage
VB	:	Valence band
v/v	:	Volume concentration
XOCs	:	Xenobiotic organic compounds
w/w	:	Weight concentration
WWTPs	:	Wastewater treatment plants
ZnO	:	Zinc oxide

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CHAPTER 1

INTRODUCTION

1.1 Research Background

The increasing population each year leads to the escalating of water demand that produces a huge amount of wastewater and greywater. It had estimated the number of the world population is about nearly 7.8 billion people in the year 2020 (Matzuk *et al*., 2020). Meanwhile, in Malaysia, the number of populations increase by year, and it was close to 32.7 million people based on the Department of Statistics Malaysia (2020). Since the population keeps increasing steadily every year, the possibility of producing wastewater is high. Recently, through RMK 11 in section 69 and 70, the Malaysian government has claimed that by 2020, 99% of the population would have sufficient and treated water.

The production of greywater coming from residential increased dramatically with respect to the population (Tsoumachidou *et al.*, 2016). It is estimated about 6 million liters of domestic greywater were produced every year in Malaysia. The huge amount of domestic greywater production can impact the quality of life if it been freely released to the waterways such as to the river and stream. Mohamed *et al*., (2018) reported that, in some rural areas of Malaysia, domestic wastewater was discharged into the river directly. Hence, of the situation needs to be handled and mitigated wisely since domestic greywater considered a complex mixture containing water together with common constituents such as xenobiotic organic compounds (XOCs) influenced by personal care products (Noman *et al*., 2019; Huang *et al*., 2017; Eriksson *et al.*, 2003). Greywater that comes from ablution, sinks, laundry, the bathroom has a concentration of XOCs influenced by pharmaceutical and personal care products (PPCPs) and offer negative effects to the human health and human

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health (Yang *et al*.,, 2017; Yang *et al*., 2008; Eriksson *et al*., 2002). Addressing these problems calls out for a tremendous amount of research to be conducted to identify new methods of purifying water preferably green method with less energy, while at the same time minimizing the use of chemicals and impact on the environment (Shen *et al*., 2012; Malato *et al*., 2009).

The continuous persistence of TCC in rivers and streams is a major issue to the environment and humankind. TCC is a topical antiseptic but can end up in the food chain, which is neither regulated nor monitored. There are several types of XOCs especially TCC that would not be degraded after conventional treatment of greywater, and due to that case, advance treatment is needed (Thelusmond *et al*., 2018; Gisi *et al*., 2016; Leal *et al*., 2010 Chu and Metcalfe, 2007).

The application of advance treatments is very important because as the concentration and number of contaminants increase, the process becomes more complicated. The problems can be attributed to catalytic deactivation, slow kinetics, low photo efficiencies, and unpredictable mechanisms (Zhou *et al*., 2018) Some promising new method in the context of pollutant abatement in water is collectively respected to photocatalytic degradation system (Hunge *et al*., 2021; Ge *et al*., 2017). Photocatalytic degradation was widely used for decontamination of water containing organic pollutants that has been classified as recalcitrant compounds (Kurniawan *et al*., 2020). These photocatalytic degradation methods such as titanium dioxide (TiO_2) photocatalysis rely on the formation of highly reactive chemical species such as TiO_2, which degrade even the most recalcitrant molecules into biodegradable compounds (Wafi *et al*., 2021; Gulyas *et al*., 2009).

TiO_2 photocatalysis is gradually developed as an affordable, effective, environmentally friendly, reusable, and sustainable technology in wastewater and greywater treatment (Priyanka *et al*, 2020). Titanium (Ti), well known as the world's fourth most abundant metal and the ninth most abundant element, was discovered in 1791 by Reverend William Gregor in England (Kumar & Misra, 2020; Li, 2013). Pure TiO_2 does not occur in nature; it is primarily found in various minerals like rutile, ilmenite, leucoxene, anatase, brookite, perovskite, and sphene, and titanates and many iron ores as well (Li, 2013). TiO_2 remains one of the most promising materials due to its high oxidation efficiency, nontoxicity, high photostability, chemical inertness, and environmentally friendly nature (Reddy *et al*., 2020; Zhang *et al*., 2010; Di *et al*., 2007). Another advantage of TiO_2 is its low cost, owing to the

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abundance of Ti estimated 0.44% of Earth's crust (Westerhoff *et al*., 2011; Robert, 2007). Worldwide reserves over 600 million tons, the annual production of Ti metal is approximately 90,000 tons, and the annual production of TiO_2 is around 4.3 million tons (Westerhoff *et al*., 2011). Previous research has shown that the TiO_2 semiconductor is an excellent photocatalyst that can perform under UV irradiation to mineralize a large range of refractory organic pollutants, for examples antibacterial, surfactant, herbicides, dyes, pesticides, and phenolic compounds, among others (Mao *et al*., 2017; Daghrir *et al*., 2013).

However, the major drawback of such TiO_2 nanoparticle is the random pathway of photoinduced electrons during photocatalytic reactions, which eventually lead to the recombination through trapping or de-trapping of electron/hole pairs as well as a longer electron-transporting time in the particulate TiO_2. In addition, the probability of recombination losses of photoinduced charge carriers would be increased significantly due to the presence of defects or trapping sites, more grain boundaries, light scattering, and disordered contact areas (Malakootian *et al*., 2020; Mohamed and Rohani, 2011; Yan and Zhou, 2011; Lei *et al*., 2010; Sun *et al*., 2010; Kasuga *et al*., 1998). Meanwhile, it was found that TiO_2 nanotubes could eliminate the reusability of photocatalyst issues and ease the filtration procedure after photoreaction (Zhu *et al*., 2020; Sun dan He, 2010; Ghicov and Schmuki, 2009). In order to further maximize the specific surface area of TiO_2 nanotubes for better photons absorption from illumination, design and development of TiO_2 based nanostructure assemblies have gained significant interest and triggered enormous effort in physics, chemistry, and material science (Yan & Zhou, 2011; Ge *et al*., 2017; Su *et al*., 2011; Sun *et al*., 2010; Ghicov & Schmuki, 2009; Chen & Mao, 2007; Grimes, 2007). It is because TiO_2 nanotubes are becoming more popular due to their high photocatalytic activity for various types of degradation (Zhou *et al.*, 2018; Lai *et al*., 2014).

Highly ordered and vertically oriented TiO_2 nanotubes can be fabricated by anodization of Ti metal foil under suitable electrolyte and processing conditions. TiO_2 nanotubes are gained after the process that initially involves the formation of the barrier layer and followed by rather well defined nano-porous structure (Mohan *et al*., 2020; Lai & Sreekantan, 2011; Macak *et al*., 2007; Grimes, 2007; Macak & Schmuki, 2006). The Ti metal foil was used as a substrate for TiO_2 nanotubes to grow. The amorphous TiO_2 nanotubes can be obtained at the end of the anodization

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process. Other than that, to extend the spectral response of TiO_2 nanotubes into the visible region and to enhance its photocatalytic activity, several strategies have been developed. Coating pure TiO_2 nanotubes with metal cations such as zeolite is one way for sensitizing TiO_2 to visible light (Zhang *et al*., 2018; Zhang *et al*., 2012; Wang *et al*., 2006). The zeolite used as an adsorbent, an ion exchanger, and a catalyst zeolite significantly increased the adsorption capacity of the composite photocatalyst, resulting in a decrease in the concentration of intermediates that are desorbed from the photocatalyst surface and released to the gas phase during the advance oxidation processes (AOPs) (Koohsaryan & Anbia, 2016; Mao *et al*., 2017). Since zeolite has potential in assisting the performance of TiO_2 nanotubes, this route promotes the use of the main part of the solar spectrum and also to ensure the charge traps to keep electron-hole photogenerated separate (Nguyen *et al*., 2020; Prakash *et al*, 2019; Faraji & Mohaghegh, 2016). Therefore, this study aims to establish the TiO_2 nanotubes coated with Zeolite (TNTs/Zeo) as a photocatalyst to enhance the photocatalytic performance of TCC degradation in artificial bathroom greywater (ABGW).

1.2 Problem Statement

TCC is acknowledged as one of the hazardous compounds and could lead to negative impact to the environmental and human health. TCC effects has been reported on a variety of mammals, for example rats and rabbits. The report showed the reproduction and survival rates of offspring decrease in rats, and it is proportional to the elevated TCC levels (Li *et al*., 2018). TCC is also known to cause methemoglobinemia in human. It can also cause cancer and baby blue syndrome, a condition of decreasing oxygen-carrying capacity of hemoglobin in babies leading to death (Bomar *et al*., 2017). Due to its side effects, in 2013 FDA has proposed the rulemaking regarding allowable TCC concentration in products and in September 2017, FDA issued a final rule of banning the application of TCC in various personal care products. Nevertheless. In 2018, European Chemical Agency and British Environment Agency have reported TCC concentration in surface water has been found at risk level and could affect the aquatic life at the level of 3 μg/L to 10 μg/L of concentration that could end up in the food chain, which is neither regulated nor monitored. In addition, the information of TCC in Malaysia is still lacking in many ways, such as the actual detected of TCC concentration in bathroom greywater effluent in entire nation and due to that, there is yet no official standard and regulation provided by government of Malaysia in order to monitor TCC concentration in water bodies.

The continuous persistence of TCC in water body is a major concern to the environment and human health due to its toxicity and hazardous effects. TCC in greywater are not only recalcitrant but can also be adsorbed on solid particles via electrostatic forces. TCC also has a strong chemical structure which makes TCC as a reactive compound. Hence, it complicates the degradation process of the compounds using biological water treatment or other conventional water treatment processes (Rogé *et al*., 2017; Rahman *et al*., 2009; Nakada *et al*., 2007; Eyers & Fantroussi, 2004;). The mentioned issue has led to the advance oxidation processes (AOPs) to improve the removal of TCC in greywater such as photocatalytic degradation technique (Lazar *et al*., 2012). Photocatalytic degradation is one of promising technique to degrade TCC according to its potential to degrade recalcitrant compounds. Other than that, in AOPs, the photocatalytic degradation can be customized by various selections of photocatalyst based on the characteristic of targeted compound or pollutant.

TiO_2 is one of general and effective photocatalyst applied in photocatalytic degradation as the main role in the process. The position of titanium ions creates favorable reaction condition against organic compounds. Its strong resistance to chemical breakdown, high stability towards photo-corrosion, non-toxicity, low cost, long term stability are the main factors of photocatalyst selection. However, the TiO_2 photocatalyst alone inadequate to degrade recalcitrant compounds especially TCC due to TiO_2 photocatalyst has high recombination of electron-hole where could decrease the photocatalytic performance activities. In consequences, it is clear that unmodified TiO_2 usually needs a solution to undertake practical applications of industrial and environmental interest (Daghrir *et al.*, 2013). This could lead to the loss of some of the operation effects. Therefore, the study intends to propose modification on the catalyst semiconductor, which requires metal cation (zeolite) as precursor to be coated on TiO_2 nanotubes to enhance the photocatalyst activity and the photocatalytic degradation of TCC in water since zeolite has the ability to separate the recombination of electron-hole. Zeolite is used as an absorbent, ion

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PTTA UTHM PERPUSTAKAAN TUNKU TUN AMINAH

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