REMOVAL OF BISPHENOL A FROM WATER VIA VISIBLE LIGHT-DRIVEN PHOTOCATALYTIC DUAL LAYER HOLLOW FIBER MEMBRANE

ROZIANA BINTI KAMALUDIN

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DEDICATION

This thesis is dedicated to my parents, lecturers, family and friends who taught me that the best kind of knowledge to have is that which is learned for its own sake and even the largest task can be accomplished if it is done one step at a time.

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ABSTRACT

In recent years, the level of bisphenol A (BPA) detected in various water sources, foods and beverages has gained media attention. The presence of BPA in the environment is highly potential to contaminate the environment and can cause various adverse affects to human and aquatic life. Photocatalysis in the presence of titanium dioxide (TiO_2) is one of the promising options which offer complete degradation of organic contaminants even at low concentrations. However, TiO_2 only displays its photocatalytic effect under ultraviolet (UV) light irradiation and is associated with difficulties of recovery after treatment process. Therefore, the present study is conducted to investigate the efficiency of visible light-driven photocatalytic dual layer hollow fiber (DLHF) ultrafiltration (UF) membrane for the removal of BPA from contaminated water and its detrimental effects by using an *in-vivo* model. Visible light-active (VLA) nitrogen-doping TiO_2 (N-doped TiO_2) was successfully prepared via simple, energy and cost saving sol-gel method. Then, N-doped TiO_2 was further incorporated into DLHF membrane with different N-doped TiO_2/ polyvinylidene fluoride (PVDF) weight ratio (0.2, 0.5 and 0.7) via co-extrusion spinning method. The photocatalytic membranes were characterized for their morphology, particles distribution, surface roughness, crytallinity and light absorption spectra. The photocatalytic activity was measured by photocatalytic degradation of BPA in contaminated water under visible and UV light irradiations in comparison with the commercial TiO_2. This is followed by investigating the effects of untreated-BPA and treated- BPA water on the tight junction protein. The expressions of Claudin-2, -3 and -4 were measured quantitatively and qualitatively via western blotting (WB), haematoxylin and eosin (H&E) and immunohistochemistry (IHC) staining. Excellent photocatalytic activity with more than 90% of BPA removal was achieved in the presence of N-doped TiO_2 DLHF under visible and UV light irradiation. The removal of BPA was 80% higher in comparison with the commercial TiO_2. *In-vivo* study showed that the BPA exposure altered the morphology of finger-like projections of villi and changed expression level of Claudin 2, 3 and 4 proteins in the jejunum and ileum of both pregnant rats and their foetuses. Interestingly, villi and Claudins expressions were undisrupted in treated-BPA water exposed-group. The presence of VLA element in N-doped TiO_2 DLHF significantly improved the absorbance capability under visible irradiation, thus increases the photocatalytic degradation activity under visible light irradiation and effectively mitigated the effect on BPA on the ileum and jejunum of gastrointestinal tract. Altogether, successfully fabricated photocatalytic DLHF membrane using co-extrusion method has a promising potential in removing BPA to fulfill the public focus on the safety of water and their need to consume clean water.

ABSTRAK

Sejak beberapa tahun kebelakangan ini, tahap bisphenol A (BPA) yang dikesan dalam pelbagai sumber air, makanan dan minuman telah mendapat perhatian media. BPA berpotensi tinggi untuk mencemarkan alam sekitar dan menyebabkan kesan yang buruk kepada manusia dan kehidupan akuatik. Fotopemangkin dengan kehadiran titanium dioksida (TiO_2) adalah salah satu pilihan rawatan yang menjanjikan degradasi lengkap dalam merawat bahan pencemar organik walaupun pada kepekatan rendah. Walau bagaimanapun, fotopemangkin TiO_2 hanya berkesan di bawah sinaran cahaya ultraviolet (UV) dan TiO_2 telah dikaitkan dengan kesukaran pengumpulan semula zarah selepas proses rawatan. Oleh itu, kajian ini dijalankan untuk mengkaji kecekapan membran fotopemangkin ultraturasan gentian optik dwi-lapisan (DLHF) untuk menyingkirkan BPA dan kesan buruknya dari air dengan menggunakan model *in-vivo*. Nitrogen-doping TiO_2 (N-doped TiO_2) yang aktif di bawah cahaya nampak (VLA) berjaya dihasilkan melalui kaedah sol-gel yang mudah, menjimatkan tenaga dan kos. Kemudian, ianya dimasukkan ke dalam membran DLHF dengan nisbah berat N-doped TiO_2/polyvinylidene fluoride (PVDF) yang berbeza (0.2, 0.5 dan 0.7) melalui kaedah penyemperitan bersama. Membran fotopemangkin dicirikan dari segi morfologi, taburan zarah, kekasaran permukaan, kehabluran dan spektrum penyerapan cahaya. Aktiviti fotopemangkin membran N-doped TiO_2 diukur di bawah sinaran cahaya nampak dan UV dengan perbandingan TiO_2 komersial. Ini diikuti dengan kajian kesan BPA yang tidak dirawat dan dirawat ke atas ekspresi Claudin-2, -3 dan -4 secara kuantitatif dan kualitatif melalui western blotting (WB), pewarnaan haematoxylin dan eosin (H & E) dan immunohistochemistry (IHC). Aktiviti fotopemangkin yang efektif dengan lebih daripada 90% penyingkiran BPA direkodkan dengan kehadiran N-doped TiO_2 DLHF di bawah sinaran cahaya nampak dan sinaran UV. Penyingkiran BPA adalah 80% lebih tinggi berbanding dengan TiO_2 komersil. Dalam kajian *in-vivo*, pendedahan kepada BPA menyebabkan perubahan morfologi unjuran villi dan ekspresi Claudin -2, -3 dan -4 protein pada jejunum dan ileum tikus hamil dan fetusnya. Menariknya, tiada perubahan yang ketara terhadap villi dan ekspresi Claudins daripada kumpulan air terdedah BPA yang dirawat. Kehadiran unsur VLA dalam N-doped TiO_2 DLHF jelas telah meningkatkan keupayaan penyerapan cahaya nampak, seterusnya meningkatkan aktiviti penguraian fotopemangkinnya. Ia juga mampu mengurangkan kesan BPA pada ileum dan jejunum di saluran gastrointestinal. Secara keseluruhannya, membran fotopemangkin DLHF yang berjaya dihasilkan melalui kaedah penyemperitan bersama mempunyai potensi menyingkirkan BPA untuk memenuhi tuntutan orang ramai terhadap keselamatan air dan keperluan mereka untuk menggunakan air bersih.

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LIST OF ABBREVIATIONS

AFM	-	Atomic force microscopy
AOPs	-	Advanced oxidation processes
ATR	-	Attenuated total reflection
BET	-	Brunauer, emmet and teller
BPA	-	Bisphenol A
C_3H_7OH	-	Isopropanol
cDNA	-	Complimentary deoxyribonucleic acid
CO_2	-	Carbon dioxide
DDT	-	Dichlorodiphenyltrichloroethane
DLHF	-	Dual layer hollow fiber
DMAc	-	N,N-Dimethylacetamide
DMEM	-	Dulbecco’s modified eagle media
DMSO	-	Dimethyl sulfoxide
DWTP	-	Drinking water treatment plant
EDCs	-	Endocrine disrupting compounds
EDX	-	Energy dispersion of x-ray
EGF	-	Epidermal growth factor
EtOH	-	Ethanol
FBS	-	Fetal bovine serum
FE-SEM	-	Field emission- scanning electron microscope
FTIR	-	Fourier transform infra-red
hES	-	Human embryonic stem
HNO_3	-	Nitric acid
HPLC	-	High performance liquid chromatography
LED	-	Light-emitting diode
N-doped	-	Nitrogen doped
O_2	-	Oxygen
PBS	-	Phosphate-buffered saline
PCBs	-	Polychlorinated biphenyls
PCR	-	Polymerase chain reaction

PEG	-	Polyethylene glycol
PMSF	-	Phenylmethanesulfonyl fluoride -
PVDF	-	Polyvinylidene fluoride
RPM	-	Rotation per minute
RPMI	-	Roswell park memorial institute
RNA	-	Ribonucleic acid
SEM	-	Scanning electron microscopy
SLHF	-	Single layer hollow fiber
SPSS	-	Statistical package for the social science
STPs	-	Sewage treatment plants
$Ti(OBu_4)$	-	Titanium-n-butoxide
TiO2	-	Titanium dioxide
UV	-	Ultraviolet
VLA	-	Visible light active
WTPs	-	Water treatment plants
XRD	-	X-ray diffraction

LIST OF SYMBOLS

A	-	Membrane surface area (m^2)
Aa	-	Intergrated intensityof anatase
Ar	-	Intergrated intensity of the rutile
a.u	-	Absorbance unit
C	-	The speed of light
C_0	-	Initial concentration
C_t	-	Concentration at specific time
E	-	Band gap energy
eV	-	Electron volt
F	-	Flux
H	-	planks constant
IU	-	International unit
kN	-	Kilonewtons
kV	-	Kilovolt
L	-	Effective length of hollow fibers
L/m^2h	-	Membrane flux
MPa	-	Megapascal
Pa	-	Pascal
Ppm	-	Parts-per-million
Ra	-	Surface roughness
t	-	Time (h)
V	-	Volume of permeate at time
W	-	Watt
WR	-	Weight fraction
Wt.%	-	Weight percent
θ	-	Theta
λ	-	Wavelength (nm)

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Endocrine system is a complex network of glands that produce and release hormones that maintain body homeostasis and regulates many of the important body functions such as cell metabolism, sexual development and reproduction, as well as to regulate various organs operations. Any disruption to this balance can cause abnormal consequences in the reproduction, development, growth, or behavior of human and animals that may affect them and their offspring. Endocrine disrupting compounds (EDCs) can be defined as exogenous chemicals that may directly or indirectly interrupt the normal function of endocrine system (Chang *et al.*, 2009). EDCs either natural or synthetics (Filali-Meknassi, 2004) are detectable in the product of industry, agricultural, pharmaceuticals and household convenience (Kralchevska *et al.*, 2013). Various EDCs have been identified including bisphenol A (BPA) (Pan *et al.*, 2008), dioxin, polychlorinated biphenyls (PCB), dichlorodiphenyltrichloroethane (DDT) (Colborn *et al.*, 1996) and many more. These endocrine contaminants are suggested to have long half-lives as they are produced to maximize their effect on the designated area. Those EDCs may or may not be metabolized, or broken down into more toxic substances than the actual molecule; hence they may pass through the treatment plants unaffected and are discharged into various water sources such as surface waters, ground water and drinking water; resulting to an unfavorable trace in the environment.

Among recognized EDCs, BPA is the most studied endocrine contaminants due to their reported adverse health effects towards human and wildlife. BPA or chemical name 2,2-bis(4-hydroxyphenyl)propane, is an organic compound with two unsaturated phenol rings, highly produced synthetic monomer (Vandenberg *et al.*, 2010) which are employed in the manufacture of polycarbonate plastics and epoxy resins (Lenie *et al.,* 2008; Grasselli *et al.,* 2010; Rubin, 2011). In respect to good mechanical properties, low adsorption of moisture and thermal stability of synthetic polymers made from BPA (Huang *et al.,* 2012), BPA are so widespread used as liners in food and drinks packaging (Grasselli *et al.,* 2010), various plastic stuffs, toys as well as appears in many electronic equipment (Flint *et al.,* 2015). Therefore, the sources of BPA exposure are varying through direct contact with contaminated plastic materials, utensil and equipments, ingesting contaminated food and water, or breathing contaminated air. BPA are traced in variable water sources worldwide including wastewater effluents, sediments, sea water, ground water supplies, surface water and even filtered household water (Robinson *et al.,* 2009). In addition, BPA may leach into food, drinking water, infant formula or saliva (Grasselli *et al.,* 2010) via food and drink packaging (Stahlhut, Welshons and Swan, 2009). The level of BPA detected in foods and beverages has gained media attention over the last several years since the presence of BPA in the environment is highly potential to cause adverse ecological and human health effects. Exposure to BPA are proposed to associated with reduced sperm quality, implantation failure and miscarriage, altered thyroid hormone concentrations, diabetes, cardiovascular disease, childhood obesity and many more (Alonso-Magdalena *et al.,* 2010; Fernandez *et al.,* 2010; Midoro-Horiuti *et al.,* 2010; Signorile *et al.,* 2010). Meanhwile, *in-vitro* study done by Wetheril *et al.,* (2005) found that BPA at 1nM can modulate prostate cell proliferation and subsequently increase susceptibility of the prostate gland to hormonal carcinogenesis. These findings revealed that BPA effect are dangerous even at lower doses. Furthermore, the contamination of BPA in the aquatic environment has been found to cause adverse findings in affected aquatics life such as feminization of male sex organ, deformities and reduced body weight (Zha and Wang, 2006), disruption in the development of reproductive organs and hormone production among mammalian species (Maffini *et al.*, 2006).

The BPA and other EDCs threats to human health have led to the increasing demand of clean water sources which excites a challenge for the contaminants removal processes. Conventional treatment methods such as membrane filtration, adsorption and bioreactor based membrane may not completely remove the contaminants. Photocatalytic process as one of advanced oxidation processes (AOPs) has shown high performance for degradation of organic compounds to the harmless materials under sensible condition. In particular, photocatalysis have been used for such significant applications such as production of renewable fuels, disinfection of water and air, mineralization of organic contaminants, and synthesis of organic compounds (Stahlhut, Welshons and Swans, 2009). Heterogeneous photocatalysis accelerated by semi-conductor metal oxides such as titanium dioxides (TiO_2) is a fast-growing field and identified as a promising tool for water treatment (Kondarindes, 2015) where organic contaminants are shown to be degraded up to 90% under suspension of semi-conductor photocatalytic operation (Dalrymple, Yeh and Trotz. 2007). During the past two decades, photocatalytic removal of organic contaminants with TiO_2 has been extensively studied due to its strong oxidizing ability to break down organic compounds and produce complete mineralization (Umar and Abdul Aziz, 2013), inexpensive as compared to other material such as tungsten oxide (Stewart, 2009), perovskite (Teixeira *et al.,* 2019) and bismuth (Arthur, 2019) in addition to their ideal properties of photocatalyst such as chemical inertness and longterm chemical stability against photo- and chemical corrosion (Lee *et al.*, 2011).

Besides, superhydrophilic TiO_2 exhibits its high photocatalytic under ultraviolet (UV) light irradiation (Bhatkhande, Pangakar and Beenackers, 2001). The application of photocatalysis for the removal of contaminants from wastewater has shown satisfying evidences at degrading the contaminants and eliminating its capabilities without formation of harmful intermediates in some cases (Doll and Frimmel, 2005). However, the main shortcomings of pure TiO_2 photocatalyst is the wide band gap (Mohamed *et al.,* 2015) which is the most important properties that strongly influence the electrical and optical properties of the material. Pure TiO_2 particles have large band gap up to 3.2 eV, hence pure TiO_2 particles only displays its high photocatalytic properties under the irradiation of ultraviolet light ($\lambda \leq 387$ nm) (Pelaez *et al.,* 2012). The higher-end of UV spectrum usually required high operating costs (Chong *et al.,* 2010), which become another main disadvantages in the production of large-scale industrial photochemical synthesis. Besides that, the rate of the photocatalyst development has been constrained due to energy efficient sources of UV light have only recently become available. With regard to that, the development of heterogeneous visible light active (VLA) photocatalysts has captured much attention recently due to their nature of easy recycling and simple synthesis method. Interestingly, it exhibited high reactivity under visible light irradiation, hence greater benefit for photocatalysis application.

In the meantime, the fabrication of photocatalytic dual layer hollow fiber (DLHF) membrane has received great attention in research world. Compared to the single layer hollow fiber (SLHF) membrane, photocatalytic DLHF has huge advantages due to their compact design and high loading of photocatalyst immobilized at the outer layer of the DLHF membrane to ensure the effective separation followed by fast degradation of contaminant (Dzinun *et al.,* 2015b). Recently, polyvinylidene fluoride (PVDF) has been widely used as both outer and inner layer membrane material as they are recognized to have excellent resistance to corrosive chemicals, powerful mechanical strength, good thermal stability and ease of processing ability with common organic solvents (Lee *et al.,* 2015). Meanwhile the immobilization of an additive into polymer membrane is shown to increase the membrane pore size and porosity (Khayet, Mengual and Matsuura, 2005). To date, no studies have been reported on the degradation of BPA by visible light-driven photocatalytic DLHF membrane. On the other hand, most studies examining the impact of BPA have focused on various organs except small intestine. Although the gastrointestinal tract (GIT) is in direct contact with BPA, there are no studies on the effect of BPA on small intestine. Hence, the present study is conducted to investigate the immobilization of visible light active (VLA) photocatalyst into DLHF ultrafiltration (UF) membrane for efficient photocatalytic degradation of BPA from contaminated water as well as further investigation on the removal of its detrimental effects by using an *in-vivo* model.

1.2 Problem Statement

Visible light is abundant in nature compared to the ultraviolet and infrared, but it generally cannot be adsorbed directly by the reactant molecules to drive the reaction hence, the irradiation with lower energy, visible light and infrared for example, can only drive catalysts with narrow band gap (Chen, 2013). Recently, there is emerging interest in developing method to increase the sensitivity of TiO_2 to visible light via non-metal elemental doping due to their efficiency to lower the band gap. Among non-metal dopants nitrogen has been observed as one of the promising materials for TiO_2 lattice to induce visible absorption (Lee *et al.*, 2014) mainly because of its comparable atomic size with oxygen (Viswanathan and Krishanmurthy, 2012). Besides, nitrogen exhibits small ionization energy, eco-friendly, higher stability and simple synthesis methods (Gai *et al.*, 2012; Zhang *et al.*, 2013) for an ideal dopant material. The introduction of nitrogen into TiO_2 lattice structure is believed to narrow the band gap of TiO_2, thus provide efficient photocatalytic activity under visible light irradiation (Ruzimuradov *et al.*, 2014).

The immobilization of photocatalyst into DLHF membrane matrix would offer a great advantage in water and wastewater treatment as it can solve the recovery of the photocatalyst suspension problem in addition to simultaneous separation and degradation process of BPA. In this regard, the outer layer of DLHF membrane will provide reaction site for the photocatalytic degradation in the presence of light irradiation, while the inner membrane layer will act as separation layer. The compact design of photocatalytic DLHF will provide simultaneous degradation reaction and separation processes to be performed in a single unit. As a result, the photocatalytic DLHF membranes can be directly applied for water treatment without leaving any photocatalyst suspension in the water. In addition, greater advantages will be achieve with the presence of visible light active photocatalyst which allow the utilization of the main part of the solar spectrum, even under poor illumination of interior lighting. Interestingly, the stability of polymer membrane under visible light will be less concern as the deformation of membrane might be avoided. Previously, Dzinun *et al.*, (2017) has investigated the stability of PVDF/TiO_2 DLHF membranes under long-term UV irradiation exposure and the results revealed that some differences in FTIR peaks between new membranes and used membranes were found, suggesting that UV irradiation has some effect on the stability of the TiO_2/PVDF membrane for 30 days exposure. Based on these findings, it can be predicted that the degradation of the polymer is lesser for this one as it used visible light rather than UV light in the previous work. Altogether, the immobilization of N-doped TiO_2 into DLHF membrane matrix will produce visible light-driven photocatalytic membrane with good morphology and enhanced photodegradation activity of contaminants even under poor illumination of interior lighting with complete removal of their detrimental effects.

1.3 Research Objectives

The aim of this present study is to develop efficient photocatalytic DLHF membrane for the removal of BPA from contaminated water under visible and/or UV light irradiation.

The specific objectives of this study are:

i. To synthesize visible light active N-doped TiO_2 nanoparticle photocatalyst and characterize their morphological and physical properties.

ii. To fabricate visible light-driven photocatalytic N-doped TiO_2 dual layer hollow fibre membranes with different loadings of N-doped TiO_2 via co-spinning technique and characterize their properties and evaluate the BPA degradation in water using DLHF based photocatalytic membrane reactors under UV and visible light irradiations in comparison with commercial TiO_2-P25 DLHF.

iii. To determine the expression of claudin 2, 3, and 4 in pregnant rats and fetuses of control, untreated-BPA and treated-BPA exposed rats by Haematoxylin and Eosin staining, immunohistochemistry technique and Western Blotting analysis.

1.4 Scope of Study

The present study is conducted to investigate the removal of BPA from water by photocatalytic DLHF membranes and its toxicity effect on *in-vivo* models. In order to achieve the objectives of this research, the following scopes are outlined:

i. Synthesizing the N-doped TiO_2 nanoparticles for visible light active photocatalytic reaction via sol gel method. The calcinations and drying temperatures was fixed at

400^{o}C and 75^{o}C, respectively. The photocatalytic performance of N-doped TiO_2 was evaluated in comparison with commercial TiO_2-P25 powder by suspension photocatalytic system.

ii. Preparing the outer and inner layer dope suspension solution with suitable photocatalysts/PVDF ratios. Preparation of dope solution with PVDF/TiO_2/DMAc and PVDF/N-doped TiO_2/DMAc concentration of (15/3/82 wt.%), (15/7.5/77.5 wt.%) and (15/10.5/74.5 wt.%) for outer layer dope and PVDF/PEG/DMAc concentration of (18/5/77 wt.%) for inner layer dope.

iii. Fabricating the UV and visible light active photocatalytic DLHF membranes by dry/wet phase inversion co-extrusion technique using fixed spinning condition. The as-spun fiber membranes were subject to post-treatment, dried at room temperature and stored until further used.

iv. Characterizing the membrane morphology and properties using scanning electron microscopic (SEM), energy dispersion of X-ray (EDX) analysis, atomic force microscopy (AFM), fourier transform infrared (FTIR), X-ray diffraction (XRD), UV-VIS-NIR spectrophotometer, contact angle, porosity test, testing the mechanical stability, and water permeability test.

v. Investigating the removal efficiency of BPA by using photocatalytic DLHF membranes in presence of UV/visible light in water by using submerged photocatalytic system. The initial concentration of BPA was 10ppm. The submerged system consists of 4 U-shaped membrane modules fitted in the light visible (LED Flood Light; Model: IP66, 30 W) / UV (Vilber Laurmat, k=312 nm, 30 W) photoreactor system

vi. Investigating the detrimental effect of untreated-BPA and treated BPA water by using animal models. Thirty Sprague-Dawley pregnant rats were randomly divided into 3 groups and were administered designated treatment by oral gavage beginning on day 2 of pregnancy until 21 day of pregnancy. Jejunum and ileum of rats from each group were collected. The difference in weight and water intake of rats from each group was recorded.

vii. Investigating the expression of tight junction proteins Claudin 2, 3 and 4 in jejunum and ileum of control, untreated-BPA and BPA-treated exposed pregnant rats and fetuses qualitatively and quantitatively by Western Blotting techniques, haematoxylin and eosin staining, and immunohistochemistry techniques, respectively. Data was analysed using SPSS software package where one-way ANOVA was used. The statistically significance was set at $p<0.05$, n=3-5.

1.5 Significance of the Study

The outcome of this study will provide an early insight on the potential role of visible light-driven photocatalytic DLHF membranes as a promising technology for complete degradation and removing of BPA in contaminated water. Dual layer configuration will ensure fast treatment process due to degradation and separation process occurs in a single unit. High distribution of photocatalyst in thin outer layer surface significantly reduce almost 50% of the photocatalyst usage which eventually leads to the producting cost saving. A great advantage was achived with the usage of visible light-active photocatalyst which the photocatalytic membrane can be used even under poor indoor lighting. In addition, it could prolong the membrane lifetime due to less deformation of the membrane. In-vivo models study will outline the post-treatment toxicity effects to describe the efficiency and rationale of the newly developed sustainable filter in areas where BPA level of drinking water is high to reduce its side effect thus help people to have a better health. The information gained will strengthen the research capacity in related areas, thus might be useful in the will improve the quality of household water and the management and prevention of endocrine related diseases as well as the establishment of the potentials benefit of local product invention. More importantly, the successful outcome of this study will improve the quality of household water in Malaysia, and subsequently reduce the long-term economic burden due to various health problems, thus Malaysian economic development will be significantly enhanced.

1.6 Thesis Organization

This thesis is organized into eight chapters which describe original works on fabrication of visible light-driven photocatalytic DLHF membranes with different loading of N-doped TiO_2 nanoparticles for degradation and separation process of BPA in contaminated water and the removal of its detrimental effects.

Chapter 1 briefly introduces the issues that lead to the current study. The four objectives of the study were identified, followed by the scopes of study used to attain these objectives. **Chapter 2** describe the literature reviews includes information regarding the occurrence and toxicity of BPA and applicable treatment options for BPA removal. A comprehensive review is presented on the photocatalytic mechanism; factors are affecting the photocatalytic process and the various types of photocatalytic membrane reactors in terms of configurations and design. The review also provides the development of DLHF membranes, factor that affect the membrane morphology and stability of DLHF membranes. **Chapter 3** provides complete research frameworks. All the materials, experimental set ups, working procedures and analytical methods for synthesized of nanoparticles, fabrication of membrane, characterization techniques, and membrane performance evaluations were describe in details. All the materials and procedures for animal care and biomolecular works were also discuss in details in this chapter.

Results and discussion were elaborated in chapter four until chapter seven. **Chapter 4** explains the synthesis of N-doped TiO_2 nanoparticles by using simple sol-gel method. The morphological properties, crystallinity, and optical properties of the prepared N-doped TiO_2 were characterized and described in details. Finally, the photocatalytic activity of the prepared N-doped TiO_2 nanoparticles was evaluated by using suspension photocatalytic reactor. In **Chapter 5**, a delamination-free N-doped TiO_2 DLHF membrane with different ratio of N-doped TiO_2/PVDF membranes was successfully fabricated with by using dry wet phase inversion co-extrusion technique. Different loading of N-doped TiO_2 nanoparticles in the outer dope solutions was varied. The effect of the different loading of N-doped TiO_2 in DLHF membranes was investigated on the morphological characterizations.

In **Chapter 6**, a comparison between N-doped TiO_2 DLHF and internationally commercial TiO_2-P25 DLHF membranes was studied. Membrane morphologies and the effectiveness of N-doped TiO_2 and TiO-P25 nanoparticles distributed in each membrane respectively were investigated in details. The membranes performances on BPA degradation were evaluated using submerged membrane photoreactor. The degradation result has been compared with previous study in order to evaluate the rate of NP degradation. The intermediate products of BPA have been identified by using high performance liquid chromatography (HPLC) analysis. Finally, the degradation mechanism was proposed. The removal of BPA detrimental effect was investigated in **Chapter 7**.

The effect of untreated-BPA water and treated-BPA water on the important component of small intestinal barrier of was studied by using animal models. The expressions of tight junction proteins (TJPs) Claudin 2, 3 and 4 in pregnant mother and fetuses was measured qualitatively and quantitatively by Western blotting and immunohistochemistry staining respectively. To conclude this thesis, the general conclusions of this study and some recommendations for future works have been listed in **Chapter 8**.

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2. **Roziana Kamaludin**, Mohd Hafiz Dzarfan Othman, Siti Hamimah Sheikh Abdul Kadir, A.F. Ismail, Mukhlis A Rahman, Juhana Jaafar. (2018). Visible Light-Driven Photocatalytic N-Doped TiO_2 for Degradation of Bisphenol A (BPA) and Reactive Black 5 (RB5) Dye. Water, Soil and Air Pollution (IF : 1.769)
3. **Roziana Kamaludin**, Amir Syarifuddin Mohamad @ Puad, Mohd Hafiz Dzarfan Othman, Siti Hamimah Sheikh Abdul Kadir, Zawati Harun (2019). The Incorporation of N-Doped TiO_2 into Dual Layer Hollow Fiber (DLHF) Membrane for Visible Light Active (VLA) Photocatalytic Degradation Activity. (Polymer Testing: IF:.2.247)
4. **Roziana Kamaludin,** Zatilfarihiah Rasdi,Mohd Hafiz Dzarfan Othman, Siti Hamimah Sheikh Abdul Kadir, Nor Shafina Mohd Noor, Jesmine Khan, Wan Nor I'zzah Wan Mohd Zain, Ahmad Fauzi Ismail, Mukhlis A Rahman, Juhana Jaafar (2019). Visible-Light Active Photocatalytic Dual Layer Hollow Fiber (Dlhf) Membrane And Its Potential In Mitigating The Detrimental Effects Of Bisphenol A In Water. (MDPI Membranes : Sitescore: 3.28*)*
5. **Roziana Kamaludin,** Zatilfarihiah Rasdi,Mohd Hafiz Dzarfan Othman, Siti Hamimah Sheikh Abdul Kadir, Nor Shafina Mohd Noor (2019). Bisphenol A: Metabolic disrupting chemical – occurrence in water sources and its potential removal system. (Submitted to Environmental Research journal, IF: 5.026; *Under Review)*