FABRICATION OF SILVER MODIFIED TITANIUM DIOXIDE COATED CERAMIC MEMBRANE FOR BISPHENOL A REMOVAL VIA PHOTOCATALYTIC MEMBRANE

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ABSTRACT

Titanium dioxide (TiO_2) base polymeric membrane not only excites under UV light which can cause polymer aging in the long-term operation and limits their impact but also chemical and thermal photostability of polymeric membrane become reduce, which decrease its applications for membrane treatment. The ceramic membrane has proven to overcome these limitations because it is chemical and thermally stable and has the ability to withstand high temperature. However, the high cost of ceramic membranes limits its wide utilization. In this study, abundantly available kaolin was selected as a ceramic material because of its superior mechanical strength and very cheap price. The photocatalytic Ag/TiO_2 coated hollow fiber ceramic membrane was fabricated by phase inversion and sintering method followed by simple deposition technique for the removal of bisphenol A (BPA). To study the morphologies of Ag-doped TiO_2 nanoparticles and coated ceramic membrane, the silver loading (0.4 to 1.0g) and coating time was varied from 30 to 120 s respectively. The coating and without coating ceramic membranes were characterized in terms of morphology, crystalline structure, and pure water flux and results showed an asymmetric coated ceramic hollow fiber membrane consist of Ag/TiO_2 nanoparticles on the surface of the membranes was produced. The photocatalytic membrane was further evaluated for the photocatalytic efficiency in degradation of BPA, which is present in water. The experimental results of photocatalytic activity test showed that the (0.8g) Ag/TiO_2 photocatalyst and the membrane (120s) had a good removal efficiency. The resulted coated membrane (0.8g Ag/TiO_2, 120s) exhibited the highest BPA removal rate of 88% in 180 min under visible light.

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ABSTRAK

Membran polimer berasaskan titanium dioksida (TiO_2) bukan sahaja terangsang di bawah sinaran UV yang boleh menyebabkan penuaan polimer dalam operasi jangka panjang dan menghadkan impak mereka tetapi juga kestabilan foto kimia dan termal membran polimer menjadi berkurang, yang mengurangkan penggunaannya bagi rawatan membran. Membran seramik telah terbukti dapat mengatasi keterbatasan ini kerana ia stabil dari segi kimia dan termal dan mampu menahan suhu tinggi. Walau bagaimanapun, kos tinggi membran seramik mengehadkan penggunaannya secara meluas. Dalam kajian ini, kaolin yang boleh diperolehi dengan banyak telah dipilih sebagai bahan seramik kerana kekuatan mekanikal yang unggul dan harga yang sangat murah. Serat seramik berongga bersalut Ag / TiO_2 katalis foto dibentuk melalui penyongsangan fasa dan kaedah sintering diikuti dengan teknik pemendapan ringkas untuk penyingkiran bisphenol A (BPA). Dua parameter yang terlibat dalam fabrikasi membran seramik katalis foto dan penyediaan partikel nano dopan-Ag TiO_2 telah diubah secara sistematik untuk mengkaji kesan struktur, morfologi dan sifat membran seramik dan partikel nano. i) Pemuatan perak (Ag) diubah dari 0.4 kepada 1.0 g semasa penyediaan partikel nano TiO_2 dopan-Ag dan ii) Masa pemendapan diubah dari 30 saat hingga 120 saat semasa lapisan partikel nano pada membran seramik. Membran seramik berlapisan dan tanpa lapisan dicirikan dari segi morfologi, struktur kristal, dan fluks air tulen. Hasilnya berjaya menunjukkan penghasilan membran serat berrongga seramik bersalut secara tak-simetri terdiri daripada partikel nano Ag / TiO_2 pada permukaan membran. Membran katalis foto dinilai lebih lanjut untuk kecekapan pengkatalisan foto dalam pendegradasian BPA. Hasil eksperimen ujian aktiviti pengkatalisan foto menunjukkan bahawa katalis foto (0.8g)Ag / TiO_2 dan membran (120s) mempunyai kecekapan penyingkiran yang baik. Membran bersalut yang dihasilkan (0.8g Ag/TiO_2, 120s) mempamerkan kadar penyingkiran BPA tertinggi sebanyak 88%.

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LIST OF ABBREVIATIONS

BET	-	Brunauer-Emmett-Teller
BG	-	Band gap
BPA	-	Bisphenol A
CB	-	Conduction band
DI	-	Deionized water
EDX	-	Energy dispersive x-ray
MF	-	Micro filtration
NF	-	Nano filtration
NMP	-	n-methyl-2-pyroolidinone
OP	-	Organic pollutants
PCD	-	Photochemical degradation
PESF	-	Polyethr sulfone
PSf	-	Polysulfone
PU	-	Polyurethane
SEM	-	Scanning electron microscope
TBOT	-	Tetra butly titanate
UF	-	Ultra filtration
UV	-	Ultra violet
VB	-	Valance band
XRD	-	X-ray diffraction

LIST OF SYMBOLS

A	-	Surface are
Au	-	Gold
C_o	-	Initial concentration
C_t	-	Concentration at certain time
d_i	-	Intial diamter
d_o	-	Ouetr diamter
e^-	-	Electron
eV	-	Eletron volt
ε	-	Porosity
h^+	-	Hole
j	-	Flux
LC	-	Liquid chromatography
M.P	-	Melting point
M_1	-	Mass of the dry membrane
M_2	-	Mass of the wet membrane
NO_3^-	-	Nitrate ion
O_2^-	-	Super oxide radical
OH^-	-	Hydroxyl ion
Pa.s	-	Pascal second
Pd	-	Palladium
RR	-	Removal rate
V_μ	-	Intial volume
V_θ	-	Volume of the feed

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

A variety of chemical substances entering the natural environment are giving cause for concern. Various contaminants such as azo dyes, organochlorine and aromatic hydrocarbons have been detected, and a number of these chemical components are suspected of being endocrine disrupting chemicals (EDCs) (Vandenberg *et al*. 2013). Bisphenol A [2,2-bis(4- hydroxyphenyl)propane, BPA] is a raw material for polycarbonate resins and is also used as a stabilizing agent for many plastics. The BPA originating from the plastic products has been found in rivers and lakes and is a known EDC. As the most precious and renewable resource in the world, water is an important aspect of life. So achieving high water quality is a critical responsibility for the water industry(Leong *et al.* 2014). It is evaluated that about 1.2 billion peoples cannot safe the drinking water, 2.6 billion have practically no hygiene, and many of individuals passed on of serious waterborne diseases yearly(S. Khan et al. 2013).

Around 98% of potable water in Malaysia is delivered to house by rivers containing salted wastes, urban and industrial waste prior to the conventional water treatment processes(Al Aani *et al*. 2017). So domestic and industrial wastewater needs to be considered for removal of organic pollutant like BPA. The problem caused by organic pollutants can be overcome by wastewater treatment. Chemical, physical and biological processes are commonly used for water treatment(Lai *et al.*

2013). But these treatments produce some other pollutants during process, which may be more toxic than primary pollutants(Muhamad *et al*. 2016). From the last decades, water purification through membrane has secured much interest. The benefits of membrane innovation are, less energy requirement, very simple flow sheet, good effluent quality and better for environment(Khin *et al.* 2012).

On the other hand, the processes are disposed to membrane fouling effects, which lead to decrease in permeate flux(Liu *et al.* 2012). However, photocatalysis has became an ecological friendly technique to destroy many sorts of toxic and intractable contaminants. A number of semiconductor photocatalyst utilized like CdS, MnO_2, TiO_2, ZnO_2, SiO_2 etc. but TiO_2 has been observed to be the most appropriate because of its nontoxic, economical, balanced, and its high generation of oxidative hydroxyl properties (OH). Still fast recombination of electron-hole pair constraints the effectiveness of TiO_2. Moreover, deficiencies of TiO_2, such as low quantum efficiency and stability, seriously restrict its progress(Liu *et al*. 2015).Due to its large band gap(3.0-3.2 eV), TiO_2 photocatalyst only excite under ultra violet rays. Nobel metal doping like platinum (Pt), palladium (Pd), gold (Au) and silver (Ag) could narrow the band gap of photocatalyst by changing its crystallinity or causing structural defects of crystal lattice to introduce impurity or defect energy level(Rajabi *et al.* 2013),which reduce the band gap from UV region to visible region and improve its photocatalytic performance. Among the noble metals (Au, Pd, Pt, Ag) used as electron traps silver (Ag) is extremely suitable industrial application due to its low cost and easy preparation. It is practically showed that Ag doped TiO_2 enhance the productivity of TiO_2 by reducing the bad gap from UV region to visible region (Sarteep *et al.* 2016).

In recent years, photocatalysis and membrane filtration have been proposed as a means for effective wastewater reclamation treatment. For the wastewater treatment method, polymeric membranes have been utilized broadly, but under UV light chemical, thermal and photo-stability of polymeric membranes become reduces which decrease its utilization for membrane treatment (Kamoun *et al.* 2017). The unique thermal, chemical and mechanical properties of ceramic membranes give them significant advantages over polymeric membranes in many applications. Ceramic membrane has revealed larger quality as far as its heat stability, mechanical properties, biological stability, easy to clean(Mustafa *et al*. 2016). Ceramic membranes are operated in the cross flow filtration mode, which has the benefit of maintaining a high filtration rate for membrane filters compared with direct flow filtration mode of conventional filters(Sondhi *et al.* 2003). Ceramic membrane has high selectivity, longer life expectancy and high cost, which limits their applications. In this study the photocatalytic ceramic membrane has been successfully fabricated using kaolin as a main material and the removal photocatalytic efficiency by the photocatalytic coated ceramic membrane is achieved from 33% to 88% in 180 mints under visible light.

1.2 Problem Statement

Photocatalysis technology is an effective and green means for the removal of persistent organic pollutants in water (Lee *et al.* 2016). Nowadays, the most widely investigated photocatalyst is still UV responsive TiO_2 catalyst (because of higher band gap, 3.0-3.2eV). Hence, the utilization of sustainable luminous energy like visible light or sunlight is limited. Moreover, deficiencies of TiO_2, such as low quantum efficiency and stability, seriously restrict its progress(Pham and Lee 2014). So optimal band gap and improvement in the photocatalytic efficiency of TiO_2 can be obtained by modify the surface of TiO_2 through doping of metal or non-metal. Silver (Ag) has shown an enhanced electron-hole separation and interfacial charge transfer ability and, as well as the increase of the visible light excitation of TiO_2

Former studies mostly focus on preparation of TiO_2-based polymeric photocatalytic membranes under UV irradiation, which will cause polymers aging in long-time operation(Adeleye *et al.* 2016) and the chemical and thermal photostability of polymeric membrane become reduce under high temperature, which decrease its utilization for membrane treatment applications. Ceramic membranes have many advantages compared to polymeric membranes including, high mechanical strength, superior chemical resistance, long service life, low energy consumption and excellent thermostability (Zhang *et al*. 2016). From the literature, it can be deduced that different ceramic materials used for membrane fabrication produced a hollow fiber membrane with different structures and properties. In this study, kaolin was selected as a ceramic membrane material. However, limited research has been focused on the Ag/TiO_2 coated ceramic membrane structural through phase inversion method followed by dip coating method drives an interest to study the process conditions effects towards the structure and properties (Bet-moushoul *et al.* 2016). Since Ag/TiO_2 nanoparticles makes drastically improvement in photocatalytic efficiency, because it shifts the ultraviolet region of TiO_2 to the visible region. Thus, using Ag/TiO_2 coated ceramic membrane, aging and fouling of membrane can be minimized and doping of metal into photocatalyst gives high surface area for the photocatalytic reaction and enhanced the photocatalytic activity.

1.3 Objectives of the Work

The aim of this research study is to fabricate, characterize and evaluate the performance of photocatalytic ceramic hollow fiber membrane made from kaolin by combined phase inversion and dip coating method for removal of bisphenol A in water. Different loading of silver was employed to achieve the Ag-doped TiO_2 nanoparticles. At the later stage, this study also aimed to further enhanced the photocatalytic efficiency of ceramic membrane by coating the Ag-doped TiO_2 nanoparticles on the surface of ceramic membrane. The Ag-doped TiO_2 coated ceramic membrane could effectively remove the BPA from the water. To achieve this aim, the following objectives were carried out:

1) To investigate the influence of Ag loading during preparation of Ag-doped TiO_2 nanoparticles on the structural and morphological properties of TiO_2 photocatalyst
2) To study the effect of coating time during coating of Ag-doped TiO_2 nanoparticles onto the ceramic membrane on the morphological and structural properties of coated ceramic membrane.

1.4 Scope of the Work

The following scope of the work is carried out to achieve the objectives

1) Preparing the Ag/TiO_2 nanoparticles as a photocatalyst source using liquid impregnation method. The amount of Ag was varied from 0.4g, 0.6g, 0.8g and 1.0g.
2) Preparing the ceramic suspensions for spinning process using ball mill at kaolin loading (40%) and kaolin to polymer ratios (1:8)
3) Fabricating ceramic hollow fiber precursor by dry-wet phase inversion method at 10 cm air gap.
4) Sintering the ceramic hollow fiber precursor at 1250^{o}C using high temperature furnace.
5) Depositing nanoparticles on ceramic membrane using dip coating technique by controlling deposition time (30, 60, 90, 120 s)
6) Investigating the morphology of coated ceramic hollow fiber membrane and Ag-doped TiO_2 nanoparticles using Field Emission Scanning Electron Microscopy (FESEM) analysis.
7) Analyzing the effect of silver loading of on the surface area of TiO_2 by Brunauer-Emmett-Teller (BET) analysis.
8) Evaluating the band gap of coated ceramic membrane and Ag-doped TiO_2 nanoparticles using UV-Vis spectrophotometer.
9) Analyzing the crystallinity of coated membrane and nanoparticles by X-ray diffraction (XRD) analysis.
10) Analyzing the point ID and mapping of both coated membrane and Ag-doped nanoparticles by energy dispersive X-ray analysis (EDX).
11) Evaluating the permeability of coated ceramic hollow fiber membrane using water permeability test.
12) Evaluating the performance of Ag/TiO_2 coated hollow fiber ceramic membrane using synthetic bisphenol A (BPA, 10ppm) as a test substance in a membrane photocatalytic reactor using visible light (100W).

1.5 Significance of Study

The study is significant to be carried out. EDCs are contaminant that harmful and give long term effect to human and wildlife. The proposed photocatalytic membrane process is one of the promising technique for EDCs removal. With the careful finding the morphology and structure of membrane might provide the promising solution in removal of BPA. This research is not only give an overview of the effect of process conditions during modification of ceramic membrane via simple dip coating method on the morphology of ceramic membrane but also present enhanced removal efficiency of BPA.

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