POLYETHERSULFONE-SILICON DIOXIDE HOLLOW FIBER ULTRAFILTRATION MEMBRANE FOR REMOVAL OF BISPHENOL A

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Specially dedicated to;

my beloved parents

for raising me up to become a useful person and giving me ample education for what I have achieved today

my siblings

for supporting the path that I choose for my future career and help me in time of need

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ABSTRACT

Extensive utilization of bisphenol A (BPA) in industrial production of polycarbonate plastics has led to frequent detection of BPA in water sources. It is a main concern among society as BPA is one of the endocrine disruption compounds that can cause hazard to human health even at extremely low concentration exposure. In view of this, polyethersulfone-silicon dioxide (PES-SiO_2) hollow fiber ultrafiltration (UF) membrane was developed in this study and used as advanced water treatment process to tackle the problem of BPA. The membrane is composed of PES as main membrane forming material, dimethylacetamide as solvent and two additives i.e. SiO_2 nanoparticles and polyvinylpyrrolidone. The SiO_2 nanoparticles were initially modified using sodium dodecyl sulfate (SDS) prior to blending into PES dope solution at different loadings (0, 1, 2 and 4 wt.%). The control PES and PES-SiO_2 membranes were fabricated via dry-jet wet spinning process. The modified SiO_2 nanoparticles and the membranes were characterized before the membrane performances were tested in filtration and batch adsorption study using water sample collected from a water treatment plant in Skudai, Johor. The transmission electron microscope results reveal that the surface modification of SiO_2 nanoparticle using SDS was able to reduce agglomeration effect between the nanoparticles. The PES-SiO_2 membranes improved mechanical, thermal, and hydrophilicity property compared to control PES membrane. The membranes also displayed higher water flux and BPA removal, owing to the presence of silanol and siloxane bonding groups which contribute to the BPA removal via adsorption mechanism. The blending of 2 wt.% SiO_2 shows the highest membrane performances, recording 73.3 L/m^2.h.bar water flux and 86% BPA removal. This membrane also shows 53 μg/g BPA adsorption capacity that is in accordance to the pseudo-second-order kinetic model. Therefore, the membrane was selected for further studies under the effect of natural organic matter (NOM) and operating parameters towards removal of BPA. The presence of NOM exhibited negative impacts on the water flux and BPA removal due to membrane fouling and competition for adsorption site with BPA. The results for the effects of operating parameters demonstrated that promising BPA removal at 90-96% could be achieved. Furthermore, backwash cleaning of the membrane was able to recover more than 80% of BPA removal after three consecutive cycles of filtration. The optimization process of developed model via historical data design of research surface methodology (RSM) on the other hand, had predicted the optimum conditions for BPA removal were at the pressure of 1 bar, pH 7, 10 µg/L BPA concentration, and filtration time of 10 min that correspond to 99.61% BPA removal. In conclusion, the developed PES-SiO_2 hollow fiber UF membrane system was found to have high potential for BPA removal and application in water treatment process.

ABSTRAK

Penggunaan meluas bisphenol A (BPA) dalam pengeluaran industri plastik polikarbonat telah membawa kepada kekerapan pengesanan BPA di dalam sumber air. Ini merupakan kebimbangan utama di kalangan masyarakat kerana BPA adalah salah satu sebatian gangguan endokrin yang boleh menyebabkan bahaya kepada kesihatan manusia walaupun dengan pendedahan kepekatan yang sangat rendah. Berikutan ini, membran turasan-ultra (UF) gentian berongga polietersulfon silikon dioksida (PES-SiO_2) telah dihasilkan dalam kajian ini dan digunakan sebagai proses rawatan air lanjutan untuk menangani masalah BPA. Membran tersebut terdiri daripada PES sebagai bahan utama membentuk membran, pelarut dimetilacetamid dan dua bahan tambahan iaitu nanopartikel SiO_2 dan polivinilpirolidon. Nanopartikel SiO_2 pada mulanya dimodifikasi menggunakan natrium dodesil sulfat (SDS), sebelum dicampurkan ke dalam larutan dop PES pada kandungan yang berbeza (0, 1, 2 dan 4 wt.%). Membran PES kawalan dan PES-SiO_2 telah difabrikasi melalui proses putaran jet-kering basah. Nanopartikel SiO_2 yang diubahsuai dan membran tersebut dipercirikan sebelum prestasi membran diuji dalam kajian penapisan dan penjerapan berkumpulan menggunakan sampel air yang diambil daripada loji rawatan air di Skudai, Johor. Keputusan mikroskop pancaran elektron menunjukkan bahawa modifikasi permukaan nanopartikel SiO_2 menggunakan SDS dapat mengurangkan kesan penggumpalan antara nanopartikel. Membrane PES-SiO_2 tersebut mempunyai ciri-ciri mekanikal, terma, dan hidrofilik yang lebih baik daripada membran PES kawalan. Membran tersebut juga menunjukkan fluks air dan penyingkiran BPA lebih tinggi berbanding membran PES kawalan, oleh kerana kehadiran kumpulan ikatan silanol dan siloksan yang menyumbang kepada penyingkiran BPA melalui mekanisme penjerapan. Campuran 2 wt.% SiO_2 menunjukkan prestasi membran tertinggi, mencatatkan 73.3 $L/m^2.h.bar$ fluks air dan 86% penyingkiran BPA. Membran ini juga menunjukkan 53 μg/g kapasiti penjerapan BPA yang selaras dengan model kinetik pseudo-tertib kedua. Oleh itu, membran tersebut telah dipilih untuk kajian lanjut mengenai kesan bahan organik semula jadi (NOM) dan parameter operasi terhadap penyingkiran BPA. Kehadiran NOM menunjukkan kesan negatif kepada fluks air dan penyingkiran BPA kerana pengotoran membran dan persaingan untuk kawasan penjerapan dengan BPA. Keputusan bagi kesan parameter operasi menunjukkan bahawa penyingkiran BPA yang menjanjikan 90-96% boleh dicapai. Selanjutnya, pencucian basuhbalik membran mampu memulihkan lebih daripada 80% penyingkiran BPA selepas tiga kitaran penapisan berturut-turut. Proses optimisasi model yang dihasilkan melalui reka bentuk data sejarah kaedah permukaan penyelidikan (RSM) sebaliknya, telah meramalkan keadaan optimal bagi penyingkiran BPA pada tekanan 1 bar, pH 7, 10 μg/L kepekatan BPA, dan masa penapisan 10 min yang bersamaan dengan 99.61% penyingkiran BPA. Secara kesimpulan, membran UF gentian berongga PES-SiO_2 yang dihasilkan didapati mempunyai potensi yang tinggi untuk penyingkiran BPA dan aplikasi dalam proses rawatan air.

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LIST OF ABBREVIATIONS

AFM	-	Atomic force microscopy
$AgNO_3$	-	Silver nitrate
Al_2O_3	-	Aluminum oxide
ANOVA	-	Analysis of variance
AOPs	-	Advanced oxidation processes
ATR	-	Attenuated total refection
BET	-	Brunauer-Emmett-Teller
BJH	-	Barrett-Joyner-Halenda
BPA	-	Bisphenol A
CAS	-	Conventional activated sludge
COD	-	Chemical oxygen demand
cSMM	-	Charged surface modifying macromolecule
DMAc	-	*N,N*-dimethylacetamide
DOC	-	Dissolved organic compound
DO	-	Dissolved oxygen
DSC	-	Differential scanning calorimetry analyzer
EDC	-	Endocrine disruptor compounds
EDX	-	Energy Dispersive X-Ray
FRR	-	Flux recovery rate
FTIR	-	Fourier transform infrared spectroscopy
HA	-	Humic acid
HEMA	-	2-hydroxyethyl methacrylate
HPLC	-	High performance liquid chromatography
MBR	-	Membrane bioreactor
MF	-	Microfiltration
MgO	-	Magnesium oxide

MWCO	-	Molecular weight cut off
NF	-	Nanofiltration
NIPS	-	Nonsolvent induced phase separation
NOM	-	Natural organic matter
PES	-	Polyethersulfone
PES-SiO_2	-	Polyethersulfone-silicon dioxide
PVP	-	Polyvinylpyrrolidone
RO	-	Reverse osmosis
RSM	-	Response surface methodology
SAJ	-	Syarikat Air Johor
SDS	-	Sodium dodecyl sulfate
SEM	-	Scanning electron microscopy
SiO_2	-	Silicon dioxide
SPM	-	3-sulfopropyl methacrylate
STP	-	Sewage treatment plant
TDI	-	Tolerable daily intake
TEM	-	Transmission electron microscopy
TGA	-	Thermogravimetric analyser
TiO_2	-	Titanium dioxide
TOC	-	Total organic carbon
UF	-	Ultrafiltration
WTP	-	Water treatment plant
WWTP	-	Waste water treatment plants
ZnO	-	Zinc oxide
ZrO_2	-	Zirconium dioxide

LIST OF SYMBOLS

A	-	Effective area of the membrane (m^2)
$A_{p,j}$	-	Pore area (m^2/g)
A_{sp}	-	Cross sectional of spinneret (cm^2)
β_i	-	Coefficients of the linear parameters (dimensionless)
β_{ii}	-	Coefficients of the quadratic parameter (dimensionless)
β_{ij}	-	Coefficients of the interaction parameters (dimensionless)
β_0	-	Constant term
C	-	Constant (µg/g)
C_0	-	Initial concentration of the BPA solutions (µg/L)
C_f	-	Concentration of feed (mg/L) and (µg/L)
C_p	-	Concentration of permeate (mg/L) or (µg/L)
C_t	-	Concentration at the time t (µg/L)
D_p	-	Density of polymer ($0.37 g/cm^3$)
D_w	-	Density of water ($0.998 g/cm^3$)
J_c	-	Pure water flux of membrane after cleaning (L/m^2 h)
J_0	-	Pure water flux of membrane before cleaning (L/m^2 h)
J_w	-	Water flux (L/m^2 h)
k	-	Number of variables (dimensionless)
K_1	-	Rate constant of pseudo first order adsorption (/min)
K_2	-	Rate constant of pseudo second order adsorption (g/µg/min)
K_{ow}	-	Octanol-water partition coefficient (dimensionless)
K_p	-	Rate constant of intraparticle diffusion model ($µg/g/min^{1/2}$)
l	-	Membrane thickness (m)
m	-	Mass of the membrane fiber (g)

m_a	-	Amount of the BPA adsorbed to the membrane fibers (μg/g)
m_d	-	Amount of the BPA desorbed to the solution (μg/g)
P	-	Equilibrium pressure of adsorbates at the temperature of adsorption (mmHg)
P_0	-	Saturation pressure of adsorbates at the temperature of adsorption (mmHg)
pK_a	-	Dissociation constant (dimensionless)
ΔP	-	Trans-membrane pressure (Pa)
Q	-	Volume of the permeate water per unit time (m^3/s)
q_e	-	Adsorbed BPA amount at the equilibrium (μg/g)
q_t	-	Adsorbed BPA amount at time (μg/g)
R	-	Removal or Rejection (%)
R_a	-	Surface Roughness (nm)
R_d	-	Desorption ratio (%)
R_f	-	Fouling resistance (/m)
R_m	-	Intrinsic membrane resistance (/m)
R_t	-	Hydraulic resistance (/m)
R_t	-	BPA removal (%)
r_k	-	Radius of the inner capillary (Å)
r_m	-	Pore radius (μm)
r_p	-	Pore radius (Å)
t	-	Time (h)
T_g	-	Glass transition temperature (°C)
Δt_n	-	Change in the statistical thickness (Å)
V	-	Adsorbed gas quantity (cm^3/g)
V	-	Permeate volume (L)
V_m	-	Mono layer adsorbed gas quantity (cm^3/g)
$V_{p,n}$	-	Pore volume (cm^3/g)
W_d	-	Dry sample weight (g)
W_w	-	Wet sample weight (g)
x_i	-	Variables (dimensionless)

Greek letters

ε	-	Residual associated to the experiments (dimensionless)
ρ	-	Porosity of membrane (%)
μ	-	Water viscosity (mPa.s)

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CHAPTER 1

INTRODUCTION

1.1 Research background

Water is one of the most crucial elements on the earth where over hundred-thousand gallons of water are consumed by the general population across the world on a daily basis (Qu *et al.*, 2013). Owing to the increasing world population, there is a strong growing demand for potable water (Zhao *et al.*, 2013a; Chen *et al.*, 2011). Nevertheless, there has been an issue on the safety of drinking water concerning the frequent detection of emerging pollutants that are characterized as bioactive and persistent chemicals originating from diverse sources (Benner *et al.*, 2013).

Bisphenol A (BPA) is one of the emerging pollutants derived from industrial products that can be found in water supplies at a trace level concentration (ng/L) (Santhi *et al.*, 2012). Even though this compound is detected at a very low concentration in the environment, its continuous discharge into the water sources might lead to adverse effects on human health (Houtman, 2010). This is because BPA is an endocrine disruptor compound (EDC) that can interrupt the endocrine system by mimicking, blocking or disrupting functions of hormones in living organisms (Yuksel *et al.*, 2013).

The conventional water treatment plant (WTP) has been known to be effective in treating surface water by removing the majority of chemical and microbial contaminants. However, the WTP is not effective in removing EDCs as these compounds are not completely removed during the treatment processes

(Stackelberg *et al.*, 2007; Sodre *et al.*, 2010; Kleywegt *et al.*, 2011; Chen *et al.*, 2013). Furthermore, the demand for better water quality has required BPA to be removed from water sources, even though it only exists at extremely low concentration level (Zhang *et al.*, 2006).

Currently, physical separation process of contaminants using membrane technology has been getting a lot of attention. UF membrane particularly has great potential in water treatment process owing to its low cost and efficient performance (Yang and Chen, 2013). Among the most commonly used material for UF membrane is polyethersulfone (PES) (Huang *et al.*, 2012a; Zeng *et al.*, 2012; Shen *et al.*, 2011; Sun *et al.*, 2010; Xu and Qusay, 2004). The PES-based membrane has the advantages of high thermal resistance, wide range of pH tolerances (pH 4–10), good chlorine resistance, flexibility in membrane fabrication and high mechanical properties (Lalia *et al.*, 2013). However, PES membrane is not without drawbacks. This membrane suffers from trade-off effect between membrane rejection and water flux performance (Zhu *et al.*, 2014). In addition, the hydrophobic nature of PES membrane is prone to fouling problem that affects water flux production rate (Chen *et al.*, 2011). Hence, the membrane surface is often modified in order to improve membrane performance in long run.

The incorporation of nanoparticles into the polymeric matrices has become very popular in the field of membrane research where nanoparticles are added as inorganic additive to modify the properties of membrane. The nanoparticle of interest for this study is silicon dioxide (SiO_2), because it is less toxic compared to other nanoparticles and suitable for drinking water application (Ng *et al.*, 2013). This study is to develop PES-SiO_2 hollow fiber UF membrane system as an advanced water treatment process for removing BPA. With the incorporation of additives in the polymeric matrices, the PES-SiO_2 hollow fiber UF membrane has high potential to remove BPA without compromising water flux.

1.2 Problem statement

BPA is an emerging pollutant that can be found in Malaysia river at trace concentration level. The production of BPA in the industrial sector has led to the transports of this compound to the river through wastewater discharge that eventually causes contamination in the water sources. The size of this compound that is characterized as micro-pollutant is not effectively removed by the conventional water treatment process. As a result, the production of potable water from treatment plant for human consumption has been contaminated with BPA. The exposure toward BPA is a major concern because of the health hazard effect of the compound that can interfere with endocrine system in the body.

Today, with the increasing demand of industrial sector, the discharge and accumulation of BPA in the environment is expected to worsen the quality of water sources. In addition, there are no establishments of act or guidelines on BPA limits in the water, despite of its frequent detection. The unregulated discharge and contamination of BPA should be prevented from now as it can lead to devastating impact in the future. Other alternatives water treatment process that has been utilized to overcome the problem such as chemical and biological treatment is not a feasible solution for long-term, owing to the high energy consumption and production of by-products.

Pertaining to the issues, researchers have studied the ability of physical process using commercial UF membrane to remove BPA. However, the properties and performance of commercial membrane depend predominantly on the fabrication process and membrane materials. Some commercial membranes are unable to attain high BPA removal due to the lack of interaction between the membrane surface and the targeted compound. Incorporation of additives in the polymeric matrix is one of the ways to improve the membrane performance. The modification process can create synergistic effects in the membrane, simultaneously improving mechanical strength, thermal and chemical resistance. Therefore, this research was conducted to explore the effectiveness of self-developed nanocomposite PES-SiO_2 hollow fiber UF membranes as an advanced water treatment process to remove BPA.

1.3 Objectives of the study

The aim of the research is to evaluate the performance of in-house made PES-SiO_2 hollow fiber UF membrane and its system for advanced water treatment process in removing BPA for clean water production. This can be achieved by the following specific objectives:

i. To fabricate, characterize and evaluate the best performance of PES-SiO_2 hollow fiber UF membranes for water flux and BPA removal,
ii. To determine the effects of operating conditions and backwash cleaning efficiency on the performance of developed membrane for BPA removal, and
iii. To model and predict the best BPA removal conditions using response surface methodology (RSM).

1.4 Scope of the study

The scopes of this research are divided into three stages. *The first stage* is to develop and fabricate PES-SiO_2 hollow fiber membrane by phase inversion method (dry/wet spinning technique). The dope solution of the membrane was prepared with formulated membrane materials which include a polymer, solvent, organic and inorganic additive. Different loading of inorganic additive (SiO_2) was added into the dope solution in order to assess the best content toward membrane properties. The SiO_2 nanoparticles were initially modified using sodium dodecyl sulfate (SDS) to reduce the agglomeration in PES matrices. The modification of SiO_2 nanoparticles was studied using transmission electron microscopy (TEM), zeta potential, Fourier transform infrared spectroscopy (FTIR), particle size distribution analysis, and Brunauer-Emmett-Teller (BET) method.

The second stage is to characterize and evaluate the membrane intrinsic properties and separation performances. The morphology of the membranes was characterized by scanning electron microscopy (SEM) while the surface functionalization of SiO_2 was examined by FTIR. The thermal analysis was conducted using thermogravimetric analyzer (TGA) and differential scanning calorimetry analyzer (DSC). The hydrophilicity of membranes was measured using contact angle system. The surface roughness and surface charge of the membranes were analyzed using atomic force microscopy (AFM) analysis and zeta potential analysis, respectively. The membranes were also characterized for its mechanical properties, molecular weight cut-off (MWCO), porosity, pore size, and viscosity. The membrane performances were evaluated in term of water flux, hydraulic resistance, and flux recovery rate. The effects of SiO_2 loading on the membranes were studied for BPA removal performance via filtration and batch adsorption mode. The best performing PES-SiO_2 membrane was then assessed for BPA removal in the condition with and without the presence of natural organic matter (NOM).

The third stage is to investigate the effects of operating parameters including pressure, pH, BPA concentration and backwash cleaning on BPA removal and flux performance using the best performing PES-SiO_2 membrane that was integrated into a lab scale UF membrane system. The concentration of BPA in the feed and permeate samples were analyzed using high-performance liquid chromatography (HPLC). The statistical technique of RSM was employed to model BPA removal and optimized the process.

1.5 Significance of the study

The significance of this research to the field of environmental engineering and science is the new knowledge on the application of membrane technology for an advanced water treatment process in removing emerging pollutant using customized lab scale PES-SiO_2 hollow fiber UF membrane system. Most of the current studies have explored the removal of BPA using commercial polymer-based membranes, but none has focused on the incorporation of SiO_2 nanoparticles in polymer membrane for BPA removal. Hence, this study intend to establish new knowledge on the effects of additive concentration and operating conditions for the best BPA removal using the developed PES-SiO_2 hollow fiber UF membrane.

Apart from that, the PES-SiO_2 hollow fiber UF membrane system can improve the water quality in conventional water treatment process and prevent the contamination of BPA in drinking water sources as the composite membrane is able to remove BPA and produce high water flux. Therefore, the main health hazard of BPA carcinogenic effects from daily water consumption can be minimized. Furthermore, the design of the lab scale PES-SiO_2 hollow fiber UF membrane system is important for a full-scale study in pilot plant process as the system can be proposed to the waterworks industry in Malaysia that seeks for a reliable water treatment process with low operating and maintenance cost.

In general, this study provides a complete view on the possibility of the application of low-pressure membrane system as an advanced treatment in water treatment plant and suggests the reliable solutions to enhance the process and overcome any potential problems.

1.6 Thesis outline

The body of the thesis is divided into six main chapters. Chapter one presents brief description of the research background, including the objectives, the scope of research, and the significant of research. Chapter two provides the comprehensive literature review on the occurrences and effects of BPA, as well as the water treatment process available for BPA removal. The membrane materials, fabrication process and membrane application for BPA removal were also emphasized. Chapter three shows detailed description of the research methodology carried out in this study. This is followed by the results of membrane characterization and performance evaluation in Chapter four. In Chapter five, the performances of the membrane by filtration and batch mode, as well as the effects of operating parameters on BPA removal were discussed. The modeling of BPA removal using RSM was also included in the discussion. The last chapter of the thesis is Chapter six that gives the overall concluding remarks and suggestions for future research to further enhance the potential of membrane technology for BPA removal.

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