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IMPREGNATION OF MWCNT/ZnO DUAL NANOFILLER MIXED MATRIX MEMBRANE

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IMPREGNATION OF MWCNT/ZnO DUAL NANOFILLER MIXED MATRIX MEMBRANE

by

PANG WEN YU

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TABLE OF CONTENTS

	Page
ACKNOWLEDGEMENT	ii
TABLE OF CONTENTS	iii
LIST OF TABLES	vii
LIST OF FIGURES	viii
LIST OF ABBREVIATIONS	x
LIST OF SYMBOLS	xiii
ABSTRAK	xv
ABSTRACT	xvii
CHAPTER ONE: INTRODUCTION	1
1.1. Removal of humic acid from water source	1
1.2. Problem statement	2
1.3. Research objectives	5
1.4. Scope of Study	6
CHAPTER TWO: LITERATURE REVIEW	8
2.1. Humic substances	8
2.2. Membrane technology	10
2.3. Limitations of PES ultrafiltration membranes in humic acid removal	12
2.4. PES membrane modification methods	15
2.5. Modification of PES membrane by blending/composite	19

2.5.1. Hydrophilic polymer 20

2.5.2. Amphiphilic polymer 27

2.5.3. Inorganic and nanomaterial additives 35

2.5.3.(a) Single filler membrane 35

2.5.3.(b) Dual filler membrane 51

CHAPTER THREE: MATERIALS AND METHODOLOGY 54

3.1. Materials and chemicals 54

3.2. Flowchart of experimental activities 56

3.3. Purification and oxidation of multiwalled carbon nanotube 57

3.4. Characterization of multiwalled carbon nanotube 57

3.4.1. Qualitative verification via colloidal dispersion 57

3.4.2. Elemental analysis via SEM-EDS 58

3.4.3. Functional group analysis 58

3.4.4. Carbon structure analysis 59

3.4.5. Carbon nanotube morphology 59

3.5. Membrane preparation 59

3.6. Membranes characterization 61

3.6.1. Membrane cross section and surface morphology 62

3.6.2. Membrane surface roughness analysis 63

3.6.3. Rheological properties of membrane dope solution 63

3.6.4. Membrane surface element analysis 63

3.6.5. Membrane surface functional group analysis 64

3.6.6. Porosity and Pore Size Determination 64

3.6.7. Membrane hydrophilicity and wettability analysis 65

3.6.8. Thermal stability analysis 66

3.7. Performance evaluation of the fabricated membrane 66

3.7.1. Preparation of humic acid feed solution 66

3.7.2. Humic acid concentration measurement 67

3.7.3. Permeation system 67

3.8. Membrane fouling evaluation 69

3.9. Membrane antibacterial properties evaluation 71

3.9.1. Bacterial culture and concentration measurement 71

3.9.2. Membrane antibacterial test 72

CHAPTER FOUR: RESULT AND DISCUSSION 73

4.1. Characterization of functionalized multiwalled carbon nanotube (FCNT) 73

4.1.1. Colloidal dispersion for functionalization validation 73

4.1.2. Element analysis by EDS 74

4.1.3. Functional group analysis by FTIR 75

4.1.4. Carbon structure analysis 76

4.1.5. Carbon nanotube morphology 78

4.2. Preliminary study of dual nanofiller membrane with raw CNT and ZnO 79

4.2.1. Characterization of prepared membrane 80

4.2.2. Performance and antifouling evaluation of prepared membrane 89

4.3. Study of dual nanofiller membrane with FCNT and ZnO 94

4.3.1. Characterization of prepared membrane 95

4.3.2. Performance evaluation of the fabricated membrane 112

4.3.3. Antifouling evaluation of the ZnO/FCNT membrane 114

4.3.4. Antibacterial properties evaluation 120

4.3.5. Summary on dual nanofiller membrane with FCNT and ZnO 125

CHAPTER FIVE: CONCLUSION AND RECOMMENDATIONS 127

5.1. Conclusion 127

5.2. Recommendations 130

REFERENCES 132

LIST OF PUBLICATIONS

LIST OF TABLES

		Page
Table 2.1:	Summary for single filler membrane	46
Table 3.1:	Materials and chemicals used in the research	54
Table 3.2:	Composition of the fabricated membrane using raw MWCNT	60
Table 3.3:	Composition of the fabricated membrane using functionalized MWCNT	61
Table 4.1:	EDS chemical composition for raw CNT and FCNT	75
Table 4.2:	The membrane thickness, porosity and mean pore size of the fabricated membrane	85
Table 4.3:	Surface roughness parameters obtained from AFM images	86
Table 4.4:	Initial pure water flux, HA permeate flux and final pure water flux of the membranes	90
Table 4.5:	Relative flux reduction (RFR) and flux recovery ratio (FRR) of the membranes	92
Table 4.6:	Surface roughness parameter of FZ series membrane	101
Table 4.7:	Elemental analysis for membrane prepared with different ratio of ZnO and FCNT	104
Table 4.8:	Mean pore size and porosity of FZ series membrane	107
Table 4.9:	Pure water flux (PWF), permeate flux (HAF) and HA rejection for FZ0, FZ1, FZ2, FZ3, FZ4 and FZ5	112
Table 4.10:	Membrane flux, RFR and FRR of FZ0, FZ1, FZ2, FZ3, FZ4 and FZ5	116

LIST OF FIGURES

		Page
Figure 2-1:	Typical humic acid (HA) structure (Stevenson, 1994)	10
Figure 2-2:	Structure of PES monomer (Alenazi et al., 2017)	12
Figure 2-3:	Pore "open" and pore "closed" state of membrane at different pH (Zhao et al., 2011b)	34
Figure 3-1:	Overall research methodology flowchart	56
Figure 3-2:	Schematic diagram of dead-end filtration unit	68
Figure 4-1:	Colloidal dispersion of raw CNT (A) and functionalized CNT (FCNT) (B) in ethanol after 24 hr.	74
Figure 4-2:	FTIR spectra showing functional group for raw CNT and FCNT	76
Figure 4-3:	Raman spectra of the raw CNT a nd FCNT	77
Figure 4-4:	TEM image of raw CNT (A) and FCNT (B)	79
Figure 4-5:	SEM image of membrane cross-sections: a) RZ0, b) RZ1, c) RZ2, d) RZ3, e) RZ4, f) RZ5	81
Figure 4-6:	Viscosity of casting dope solution for RZ series membrane	82
Figure 4-7:	SEM image of membrane surface: a) RZ0, b) RZ1, c) RZ2, d) RZ3, e) RZ4, f) RZ5	83
Figure 4-8:	Contact angle of the fabricated membrane	88
Figure 4-9:	SEM image of membrane surface: a) FZ0, b) FZ1, c) FZ2, d) FZ3, e) FZ4, f) FZ5	96
Figure 4-10:	Magnified SEM image of FZ3 under magnification of 40,000X	97

Figure 4-11: Digital photographs of PES/MWCNT or PES/MWCNT/ZnO dual nanofiller membrane (a) top side and (b) back side 98
Figure 4-12: SEM image of membrane cross-sections: a) FZ0, b) FZ1, c) FZ2, d) FZ3, e) FZ4, f) FZ5 99
Figure 4-13: Viscosity of casting dope solution for FZ series membrane 100
Figure 4-14: Three-dimensional (3D) surface AFM images of the fabricated membrane a) FZ0, b) FZ1, c) FZ2, d) FZ3, e) FZ4, f) FZ5 102
Figure 4-15: FTIR spectra of FZ0, FZ1, FZ3 and FZ5 membrane 106
Figure 4-16: Contact angle of FZ0, FZ1, FZ2, FZ3, FZ4 and FZ5 109
Figure 4-17: TGA curve under nitrogen atmosphere for FZ0, FZ1, FZ2, FZ3, FZ4 and FZ5 110
Figure 4-18: Decomposition temperature of the fabricated membrane 111
Figure 4-19: Normalized flux of different membrane against filtration time 115
Figure 4-20: Filtration resistance (R_m, R_f, R_r, R_{ir}) of FZ0, FZ1, FZ2, FZ3, FZ4 and FZ5 118
Figure 4-21: Bacteria extracted from the membrane with dilution of 10^5 for : a) FZ0, b) FZ1, c) FZ2, d) FZ3, e) FZ4, f) FZ5 122
Figure 4-22: CFU count at the end of the experiment 123

LIST OF ABBREVIATIONS

AA	Acrylic acid
AFM	Atomic force microscopy
BSA	Bovine serum albumin
CA	Cellulose acetate
CAP	Cellulose phthalate
CFU	Colony forming unit
CNT	Carbon nanotube
DBP	Disinfectant by products
DI	Deionized
DMAc	Dimethylacetamide
E.coli	*Escherichia coli*
EDS	Energy dispersive X-ray spectroscopy
EPA	Environment Protection Agency
EPS	Extracellular polymeric substances
FA	Fulvic acid
FCNT	Functionalized carbon nanotube
FRR	Flux recovery ratio
FS	Flat sheet
FTIR	Fourier transform infrared spectroscopy
GO	Graphene oxide
HA	Humic acid
HF	Hollow fiber
HMO	Hydrous manganese oxide

HS	Humic substance
ID	Inner diameter
MBR	Membrane bioreactor
MCL	Minimum contaminant limit
MEA	Malt extract agar
MF	Microfiltration
MM	Mixed matrix
MMA-AA-VP	Poly (methyl methacrylate–acrylic acid–vinyl pyrrolidone)
MMM	Mixed matrix membrane
MW	Molecular weight
MWCNT	Multiwalled carbon nanotube
NF	Nanofiltration
NIPS	Non-solvent induced phase separation
NOM	Natural organic matter
NP	nanoparticles
OD	Outer diameter
PAN	Polyacrylonitrile
PANI	Polyaniline
PDMAEMA	Poly(N,N–dimethylamino-2-ethyl methacrylate)
PEG	Polyethylene glycol
PEO	Poly(ethylene oxide)
PES	Polyethersulfone
PES-b-PHEMA	Polyethersulfone-*block*-poly(2-hydroxyethyl methacrylate)
PP	Polypropylene
PPO	Poly(propylene oxide)

PS-b-PAA	Polystyrene-block poly(acrylic acid)
PS-b-PEG	Amphiphilic copolymer comb of PEG and polystyrene
PSf	Polysulfone
PVC	Polyvinyl chloride
PVDF	Polyninylvinylidene fluoride
PVP-b-PMMA-b-PVP	Poly(vinyl pyrrolidone)-b-poly(methyl methacrylate)-b-poly(vinyl pyrrolidone)
PWF	Pure water flux
RAFT	Reversible addition-fragmentation chain transfer
RFR	Relative flux reduction
rGO	Reduced graphene oxide
RO	Reverse osmosis
SA	Sodium alginate
SEM	Scanning electron microsope
SPC	Soybean phosphatidylcholine
SPES	Sulfonated polyethersulfone
TEM	Transmission electron microscope
TGA	Thermagravimetric analysis
THM	Trihalomethanes
TMP	Transmembrane pressure
UF	Ultrafiltration
UV	Ultraviolet
VIPS	Vapor induced phase separation

LIST OF SYMBOLS

A_m	Membrane effective area	cm^2
C_f	Feed HA concentration	mg/L
C_p	Permeate HA concentration	mg/L
d_p	Polymer density	g/cm^3
I_D	Intensity of D band	-
I_R	Intensity of G band	-
J_{HA}	Humic acid permeate flux	$L/m^2.h$
J_{WF}	Pure water flux	$L/m^2.h$
J_{WF2}	Final water flux	$L/m^2.h$
R	Ratio of intensities for D and G band	-
R_a	Mean roughness	nm
R_f	Total fouling resistance	m^{-1}
R_{ir}	Irreversible fouling resistance	m^{-1}
R_m	Membrane resistance	m^{-1}
R_q	Root mean square roughness	nm
R_r	Reversible fouling resistance	m^{-1}
R_t	Total fouling resistance	m^{-1}
R_z	Average difference in the height between the highest and the lowest point	nm
t	Filtration time	h
T_d	Decomposition temperature	-
V	Collected permeated volume	L
W_d	Dry membrane weight	g

W_w	Wet membrane weight	g
ΔP	Transmembrane pressure	Pa

GREEK LETTERS

μ	Viscosity	Pa.s
ε	Porosity	%

SUBSCIPT

d	Decomposition
f	Fouling
ir	Irreversible
m	Membrane
r	Reversible
t	Total

IMPREGNASI DWI PENGISI NANO MWCNT/ZnO MEMBRAN MATRIKS BERCAMPUR

ABSTRAK

Penapisan membran untuk penyingkiran asid humik merupakan cabaran dari segi penyumbatan membran yang berlaku melalui penyumbatan zarah asid humik dan bakteria yang terdapat dalam aliran suapan pada membran. Membran berpengisi tunggal mengalami masalah untuk mencapai sifat antisumbat dan antibakteria pada masa yang sama. Oleh itu, membran polietersulfona (PES) dwi pengisi dihasilkan melalui proses pemisahan fasa dengan mencampurkan zarah nano zink oksida (ZnO) dan tiub karbon nano dinding berlapis (MWCNT) pada pelbagai nisbah ke dalam larutan dop PES. Sebelum dicampurkan, MWCNT difungsikan (FCNT) dengan mengunakan asid nitric bagi menghasilkan kumpulan berfungsi hidrofilik hidroksil dan karboksilik pada permukan tiub. Oleh itu, kedua-dua pengisi nano tersebut digunakan untuk menghasilkan membran bersifat antisumbat dan antibakteria yang baik. Kesan sinergi kedua-dua pengisi nano akan dicirikan dari segi kelikatan larutan dop, morfologi, kekasaran permukaan, size liang membran dan keliangan membran, kehidrofilikan, kestabilan haba serta prestasi penapisan. Dapatan penyelidikan menunjukkan bahawa membran yang mengandungi dwi pengisi adalah lebih berliang berbanding dengan membran berpengisi tunggal dan membran PES yang tidak berpengisi. Di samping itu, peningkatan nisbah FCNT telah menyebabkan peningkatan kehidrofilikan membran. Penambahan kedua-dua pengisi nano didapati meningkatkan kelikatan larutan dop dan mengurangkan kekasaran permukaan membran. Fluks resapan meningkat dengan peningkatan nisbah FCNT. Apabila pengisi nano pada membran didominasi oleh FCNT (nisbah melebihi 0.5), penolakan asid humik (HA)

tidak terjejas walaupun keluasan liang membran meningkat. Dapatan juga menunjukkan bahawa tanpa tindakbalas fotobermangkin, fungsi antibakteria disumbangkan oleh ZnO adalah dengan fungi antirekatan dan bukannya perencetan bakteria. Dari semua membran yang dihasilkan, FZ3 dengan nisbah FCNT dan ZnO yang sama dapat mencapai prestasi optima dengan fluks air tulen sebanyak 89.66 $L/m^2.h$ dan penolakan HA sebanyak 93.21%. Berpandukan pada perintang kotoran dan kurang pelekatan bakteria pada permukaan membran, FZ3 merupakan membran dwi pengisi bersifat antisumbat dan antibakteria yang terbaik.

IMPREGNATION OF MWCNT/ZnO DUAL NANOFILLER MIXED MATRIX MEMBRANE

ABSTRACT

Membrane filtration for humic acid removal faced challenges in term of fouling caused by solute itself and the bacteria found in the feed stream. There were a lot of studies showed great antifouling and antibacterial properties at the same time. Hence, dual nanofiller polyethersulfone (PES) membrane was synthesized via phase inversion method by blending different ratio zinc oxide (ZnO) and multiwalled carbon nanotube (MWCNT) into PES dope solution. Prior blending, MWCNT was functionalized using nitric acid to form hydrophilic hydroxyl and carboxylic group on the tube surface for dispersion. The combination of both MWCNT and ZnO was able to produce combined effect improved antifouling and antibacterial synergistic effect that does not require additional reaction or interaction between them. The synergistic effect of both nanofiller on the membrane properties was characterized in term of dope solution viscosity, morphology, surface roughness, membrane pore size and porosity, hydrophilicity, thermal stability and filtration performance. The results reveal that the membrane containing dual nanofiller was porous compared to single filler membrane and neat PES membrane. Furthermore, the increase of FCNT ratio caused the membrane hydrophilicity increased. The addition of both nanofiller was found to increase the dope solution viscosity and reduced the surface roughness of the membrane. As FCNT ratio in the membrane increased, higher permeate flux was recorded. When the nanofiller of the membrane was dominated by FCNT (i.e., ratio

more than 0.5), the humic acid (HA) rejection was not affected much even though pore size of the membrane was increased. It was also been found that without photocatalytic reaction, the antibacterial properties contributed by ZnO was antiadhesion effect rather than inhibition of the bacteria. Out of all the fabricated membranes, FZ3 (dual nanofiller membrane produced with 1wt% of equal ratio functionalized MWCNT and ZnO mixture) was able to achieve optimum performance with pure water flux of 89.66 $L/m^2.h$ and HA rejection of 93.21%. Judging from its lower fouling resistance and less bacterial adhesion onto the membrane surface, FZ3 was the most antifouling and antibacterial dual nanofiller membrane.

CHAPTER ONE

INTRODUCTION

This chapter begins with some brief introduction on membrane technology in water treatment. Then, it was followed with surface discussion about mixed matrix membrane (MMM) and their role in improving the antifouling and antibacterial properties of the membrane. Finally, the chapter is wrapped up with the problem statement, objectives and thesis organization of this research project.

1.1. Removal of humic acid from water source

Humic acid (HA) is an ancient soil-derived substance aged 50-100,000 years old that originated from vegetation in freshwater lakes or edge of marine environments as well as other water source (Laub, 2012). It been proven to be beneficial for plant growth and only caused colour problem. However, the present of it in conventional treatment processes especially chlorination can induce the formation of carcinogenic disinfectant by-product (Cowman and Singer, 1996). Therefore, humic substances were limited to value of 2.5 mg/L in drinking water according to standard STN 757111 (Barlokova and Ilavsky, 2012). Various method such as coagulation (Sudoh et al., 2015), electromagnetic treatment (Ghernaout et al., 2010), flotation (Zouboulis et al., 2003) and oxidation processes (Matilainen and Sillanpää, 2010) been employed to remove humic acid prior chlorination process. However, these methods are prone to electrode fouling, required higher operating cost and energy input (Teow et al., 2017b). This situation has led researchers to select membrane technology as the more

favourable separation method for humic acid due to its relatively simple operation with lower cost and high efficiency. However, the major concern surrounding the usage of UF membrane for HA removal was the fouling of the membrane due to HA deposition and adsorption. Since, the HA removal is done prior to chlorination or the disinfection step, biofouling can also be another source of membrane fouling. Looking into this situation, extensive researches been carried out by researcher to improve the membrane resistance both toward HA and bacteria to make the application of UF membrane for HA feasible in future.

To improve the membrane properties, filler integration into membrane has been the preference and various researches. In this perspective, the selection of filler could be a key point to decide the membrane behaviour based on the intended separation performance. This was because different nanofiller inherited different properties and can interact differently with membrane base material. As stated Mari et al. (2017), the selection of fillers was the main challenges that decide the performance and separation characteristic of the membrane. Furthermore, integration of these filler into membrane often limited by their agglomeration due to their large surface area/particle size ratio. Agglomeration of filler could potentially degrade the membrane performance and separation efficiency (Ursino et al., 2018). Therefore, in recent researches, researches start to pay more attention on filler dispersion rather than filler selection since most filler has been well studied.

1.2. Problem statement

With increasing demand toward portable water, membrane technology emerges as versatile method to recover water from various sources. Polymeric UF

membrane is commonly made of polymer such as polyethersulfone (PES), polyvinylidene fluoride (PVDF) and polysulfone (PSf). Out of these, PES is one of the most popular polymers used for UF water treatment membrane researches. The reason being is due to their excellent thermal and mechanical properties and lower swelling tendency compared to other hydrophilic polymers. However, this anti-swelling property that come with the hydrophobicity of PES can be a major cause that made PES prone for the fouling by hydrophobic solute such as humic acid (HA). Besides solute fouling, biofouling due to attachment of bacterial onto the membrane surface can also be another major concern for membrane separation especially in case of separation of HA, since UF is done prior disinfection/chlorination step which bacterial can potentially found in the feed water. Such fouling can greatly reduce the membrane performance and reduce the membrane flux along time. Thus, reduce the lifespan of the membrane and its reusability. Moreover, as fouling occur along the time, pumping cost can also increase too. Both of this indirectly increase the membrane unit operating cost. To mitigate the problem, extensive researches been done to reduce the fouling tendency of the membrane as well as improve the antibacterial properties of the membrane. One of the most used method is the blending of nanoparticles into polymeric membrane to form composite or mixed matrix membrane (MMM).

Out of the all polymeric material, PES is one of the most popular polymers for ultrafiltration membrane study. This is due to their low commercial prize, outstanding oxidative, thermal, hydrolytic stability and good mechanical property (Zhao et al., 2013a). These properties are granted due to the present of aromatic compound which able to restrain the chain mobility (Bowen et al., 2001, Mockel et al., 1999). Despite all these advantages, the major problem of the PES membrane is due to its high hydrophobicity (Rahimpour and Madaeni, 2010)

As far as the author aware, most of the mixed matrix PES membrane produced only focused on the study of antifouling properties by model solute such as HA and bovine serum albumin (BSA) (Saraswathi et al., 2018, Teow et al., 2017a, Lavanya et al., 2019). Yet, there are fewer study of UF membrane that focus on the antibacterial properties of the membrane. One of the method was utilize silver as the nanofiller in the MMM as Ag was a bactericide nanoparticles which also able to improve the membrane surface hydrophilicity (Sawada et al., 2012). However, concern raise on its toxicity when leaching occur (Fewtrell, 2014). Hence, researchers move on to utilization of ZnO nanoparticles for antibacterial study (Chung et al., 2017). Yet, ZnO mixed matrix membrane could suffer from flux reduction if used at higher ZnO loading due to pore plugging (Ahmad et al., 2015). MWCNT also another particle that able to inhibit bacterial growth (Lohan et al., 2016), but blending MWCNT into membrane required it to be functionalized first for better dispersion (Daramola et al., 2017). Unfortunately, MWCNT will lost it bactericide activity once being functionalized (Arias and Yang, 2009). MWCNT blending into the membrane could be challenging method as raw MWCNT tend to agglomerate and hence, blending of functionalized MWCNT was done to improve it dispersion. To date, there are various methods used to functionalize MWCNT. Depending on the reaction involved, the properties functionalized carbon nanotube can be different Great oxidized surface always involves aggressive reaction condition and this always associated with serious damage toward the tube structure or rupture of carbon nanotube wall. Thus, affecting the performance of functionalize MWCNT in the membrane. Hence, functionalized MWCNT should be carefully characterized prior blending into membrane.

Currently, most of the research being done to produce mixed matrix membrane was through blending only one type of nanoparticles into the membrane

matrix. Only few papers focused to study the synergism and the membrane properties of mixed matrix membrane produced by blending two types of nanofillers. Looking into this situation, in this work, mixed matrix membrane was prepared by blending well dispersed oxidized MWCNT that can improve/maintain the membrane flux and hydrophilicity with the hydrophilic ZnO with antibacterial properties into PES membrane, producing PES membrane with both antifouling and antibacterial properties for separation of HA in long run. The combined properties contributed by both ZnO and functionalized MWCNT grant synergistic effect toward the membrane. The synergistic effect contribute by the intrinsic properties of both nanofiller membrane can be varies based on their mixture ratio as studied by Esfahani et al. (2015). The variation of this properties in turn can effectively affected the separation performance as well as the antifouling properties and antibacterial properties of the membrane. Therefore, in this study dual nanofiller membrane was done by blending different ratio of nanofiller into the membrane at the same time maintaining the overall nanofiller content in the membrane.

1.3. Research objectives

The objectives of this research are:-

1. To study the characteristics of functionalized carbon nanotubes for membrane fabrication.
2. To fabricate and characterize dual nanofiller mixed matrix membrane with different ratio of MWCNT and ZnO.
3. To investigate the performance, antifouling and antibacterial behaviour of the fabricated dual nanofiller mixed matrix membrane toward HA separation.