

THE INFLUENCE OF BIOSYNTHESIS ZINC OXIDE NANOPARTICLES BY
USING FRUIT EXTRACTS OF *ANANAS COMOSUS* TOWARDS THE
PERFORMANCE OF PSf/ZnO MEMBRANE

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Dedicated to my beloved parent

Raja Ahmad Bin Raja Ali & Hasnah Binti Hassan

“Both of you are always be my best inspiration”

Special for my siblings

Syuk, Ika, Wan, Adik

“Thanks for your support and encouragement in everything that I’ve done”

Also to all my lab mates and friends

Shaff, Alin, Jaja, Wanie, Qaiyyum, Faiz, Kak Liza,

“Keep the precious moment we shared together during the good and bad time”

Lastly, to my Supervisor & Co-supervisors

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Assoc. Prof. Ts. Dr. Hatijah Binti Basri

“Thanks for the guidance, spirit, knowledge and advices given”

May Allah SWT blessed us forever.

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ABSTRACT

Severe drinking water shortage is a significant problem worldwide and now becoming a crucial issue in the field of water purification. The main key of this work is to enhance membrane performance permeability, rejection, anti-fouling and antibacterial activity. This study seeks to discover the influenced of biosynthesis of Zinc Oxide (ZnO) nanoparticles by using fruits extracts of *Ananas Comosus* towards the characteristic, properties and performance polysulfone (PSf) mixed matrix membrane. The biosynthesis ZnO, (ZnO (P)) nanoparticles powder was successfully synthesized by using fruit extracts of *Ananas Comosus*. The characterization and performance of the ZnO (P) were conducted in this study and compared with commercial ZnO, (ZnO (C)). In the beginning, the purity and crystallinity of the ZnO (P) powder was determined by using X-ray diffraction analysis (XRD) that showed the optimum synthesized composition peak was matched well with the ZnO (C) with crystallite size in the range of 12.2nm. The bandgap energy value of the ZnO (C) and ZnO (P) was 2.20 eV and 3.17 eV respectively which in line with the photocatalytic activity as many as 66.4% and 96.2% for ZnO (C) and ZnO (P) respectively in this study. The inhibition area of ZnO (P) nanoparticles powder is also relatively larger than ZnO (C) nanoparticles powder. All seven samples were successfully prepared via the phase inversion method. Obviously, the addition of ZnO nanoparticles in the PSf membrane has increased the membrane hydrophilicity property that strongly affected the membrane characteristic, property and performance. The hydrophilicity of the 3% ZnO (P) as compared to 0% ZnO and ZnO (C) increased up to 34.21% and 23.92% respectively which in line with the improvement of membrane morphology, surface roughness thus further enhanced the performance in term of permeability of the membrane. SEM results displayed that the addition of ZnO nanoparticles had enhanced the membrane porosity, reduced the tensile strength, increased in surface roughness as the additives increased up to 3% ZnO (P) which leads to enhance the antibacterial properties of the membrane. Membrane permeability was increased to 140.79% in 3% ZnO (P) compared to 0% ZnO. The antibacterial activity of the membrane with ZnO nanoparticle was improved especially with the presence of biosynthesis ZnO in 5% ZnO (P). The result also showed that the green reduction agent of ZnO had exceedingly affected the membrane characterisation and its performance. In conclusion, the addition of ZnO has increased the performance of the PSf membrane especially with the green synthesis reduction agent of *Ananas Comosus* in 3% ZnO (P).

ABSTRAK

Kekurangan bekalan air secara drastik adalah masalah besar di seluruh dunia dan kini menjadi isu penting dalam bidang pembersihan air. Kunci utama kajian ini adalah untuk meningkatkan prestasi membran dari aspek kadar penapisan air, penilaian kualiti air, penilaian anti-kotoran dan aktiviti anti bakteria. Kajian ini mengkaji mengenai kesan biosintesis zinc oxide (ZnO) partikel nano dengan menggunakan ekstrak buah daripada *Ananas Comosus* terhadap prestasi membran 'polysulfone' bersama ZnO. Partikel nano biosintesis ZnO, (ZnO (P)) telah berjaya disintesis menggunakan ekstrak buah *Ananas Comosus*. Ciri-ciri ZnO (P) dan ujian prestasinya telah diuji dan di bandingkan dengan ZnO komersial, ZnO (C). Kesan campuran polimer membran PSf yang digabungkan bersama ZnO *Ananas Comosus* juga telah dikaji dengan lebih lanjut. Ketulenan dan tahap kristal serbuk ZnO telah dikaji menggunakan pembelauan sinar-x (XRD) dengan 12.2 nm saiz kristal. Nilai tenaga yang dihasilkan oleh ZnO (C) adalah 2.20 eV manakala ZnO (P) adalah 3.17 eV dimana ianya selari dengan aktiviti 'photocatalytic' sebanyak 66.4% dan 96.2% bagi ZnO (C) dan ZnO (P) masing- masing di dalam kajian ini. Penyediaan 7 jenis sampel telah berjaya dihasilkan dengan menggunakan kaedah fasa balikan. Jelas sekali, penambahan ZnO di dalam membrane PSf telah meningkatkan sifat hidrofilik membrane seterusnya memberi kesan kepada ciri-ciri, sifat dan prestasi membran. Sifat hidrofilik bagi 3% ZnO (P) jika dibandingkan dengan 0% ZnO dan ZnO (C) telah masing- masing meningkat sehingga 34.21% dan 23.92% dimana ianya selari dengan pembaikan membran morfologi, kekasaran permukaan yang seterusnya menambahbaik prestasi aliran air tulen membran. Keputusan SEM menunjukkan dengan penambahan partikel nano ZnO telah meningkatkan tahap keliangan membran, mengurangkan kekuatan regangan membran, meningkatkan kekasaran permukaan membran, dengan penambahan aditif terutamanya 3% ZnO (P) lalu mampu meningkatkan prestasi sifat anti-bakterial membran. Ujian aliran air tulen terhadap membran juga meningkat sebanyak 140.79% di 3% ZnO (P) dibandingkan dengan 0% ZnO. Sifat anti-bakterial membran dengan kehadiran partikel nano ZnO telah ditambah baik terutamanya dengan kehadiran 5% ZnO (P). Keputusan ini juga menunjukkan bahawa ZnO oleh agen pengurangan hijau telah meningkatkan sifat dan prestasi membran. Secara konklusinya, dengan penambahan ZnO telah meningkatkan prestasi membran PSf terutamanya dengan agen pengurangan sintesis hijau dari *Ananas Comosus* dengan 3% ZnO (P).

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PTTA UTHM
PERPUSTAKAAN TUNKU TUN AMINAH

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LIST OF ABBREVIATIONS AND SYMBOLS

AFM	-	Atomic force microscope
Ag ₂ O	-	Silver oxide
AgO	-	Silver(II) oxide
Al ₂ O ₃	-	Aluminium oxide
Au		Gold
BaSO ₄	-	Barium sulphate
BSA	-	Bovine serum albumin
CA	-	Cellulose acetate
CB	-	Conduction band
CaO	-	Calcium oxide
CaCO ₃	-	Calcium carbonate
CaSO ₄	-	Calcium sulphate
CuO	-	Copper(II) oxide
DGDE	-	Diethylene glycol dimethyl ether
EDX	-	Energy dispersive X-ray diffraction
Fe ₂ O ₃	-	Iron(III) oxide or ferric oxide
Fe ₃ O ₄	-	Magnetite
FESEM	-	Field Emission Scanning Electron Microscope
F-H	-	Theory of Flory-Huggins
FRR	-	Flux recovery ratio

FTIR	-	Fourier transform infrared spectroscopy
GRAS	-	Generally recognized as safe
HA	-	Humic acid
KBr	-	Potassium bromide
LiCl	-	Lithium chloride
L-L	-	Liquid-liquid demixing
MF	-	Microfiltration
MIC	-	Minimum inhibition concentration
MgO		Magnesium Oxide
MMMs	-	Mixed matrix membranes
MWCO	-	Molecular weight cut off
NF	-	Nanofiltration
NMP	-	N-methyl-2-pyrrolidone
NO ₂	-	Nitrogen dioxide
NOM	-	Natural organic matter
NWS	-	Non-woven support
O ₂	-	Oxygen
PAN	-	Polyacrylonitrile
PEG	-	Polyethylene glycols
PEI	-	Polyetherimide
PEO	-	Polyethylene oxide
PES	-	Polyethersulfone
PI	-	Polyimide
PSf	-	Polysulfone

PSf/ZnO	-	PSf membrane with ZnO as additives
PVA	-	Polyvinyl alcohol
PVDF	-	Polyvinylidene fluoride
PVP	-	Polyvinylpyrrolidone
PWF	-	Pure water flux
RO	-	Reverse Osmosis
ROS	-	Reactive oxygen species
SEM	-	Scanning electron microscopy
SiC	-	Silicon carbide
SiO ₂	-	Silica
TEM	-	Transmission electron microscopy
TiO ₂	-	Titanium dioxide
TMP	-	Trans-membrane pressure
TM-PSf	-	Tetramethyl bisphenol-A polysulfone
TSA	-	Tryptic soy agar
UF	-	Ultrafiltration
UV	-	Ultraviolet
UV-Vis	-	Ultraviolet–visible
VB	-	Valence band
XRD	-	X-Ray diffraction
ZnCl ₂	-	Zinc chloride
Zn(NO ₃) ₂	-	Zinc nitrate
ZnO	-	Zinc oxide
ZnO-NPs	-	ZnO nanoparticles

0% ZnO	-	Pristine membrane
1% ZnO (P)	-	PSf membrane with 1wt.% ZnO
2% ZnO (P)	-	PSf membrane with 2wt.% ZnO
3% ZnO (P)	-	PSf membrane with 3wt.% ZnO
4% ZnO (P)	-	PSf membrane with 4wt.% ZnO
5% ZnO (P)	-	PSf membrane with 5wt.% ZnO
ZnO (C)	-	ZnO – Commercial
ZnO (P)	-	ZnO – Pineapple
ZrO ₂	-	Zirconium dioxide
%	-	Percentage
wt. %	-	Weight percentage
°C	-	Temperature unit in degree celcius
μ	-	unit of size in micron
a.u	-	Unit of intensity
e ⁻	-	Electron
eV	-	Unit of binding energy (electron-volt)
g	-	unit of weight (gram)
h ⁺	-	Hole
n	-	unit of size in nano
cm	-	Centimeter
nm	-	Nanometer
m	-	Meter
M	-	Mega
μ	-	unit of size in micron

bar	-	metric unit of pressure
Da	-	unit of molecular mass, Dalton (Da)
pH	-	numeric scale used to specify the acidity or basicity of an aqueous solution
PSI	-	Pounds per square inch
Q	-	Permeate volume
A	-	Membrane area
Δt	-	Time
m^3	-	unit of volume in meter
m^2	-	unit of area in meter
L	-	Litre
h^2	-	Planck's constant
y^0	-	Pseudo-steady-state permeability
J_{WF}	-	Membrane flux at the end of filtration process
C_p	-	Solute concentration in permeate stream
C_f	-	Solute concentration in feed stream
W_w	-	The weight of the membrane in wet condition
W_d	-	The weight of the membrane in dry condition
ρ_w	-	The density of the pure water at room temperature
V	-	Volume
ϵ	-	Porosity
R_z	-	Point average roughness
kPa	-	unit of pressure, pascal in kilo
eV	-	Electronvolt
rpm	-	Revolution per minute

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Membrane technology has recently gained popularity among the scientific and engineering community. A membrane that always known as a selective barrier which allows particular element or compound to pass through them such as molecules, ions or other small particles and also has a strong ability to block others unwanted materials. Membrane technology is widely applied for the purification of pharmaceuticals, water desalination, industrial effluent treatment, processing of foods, and the removal of organic compounds from aqueous streams.

The membrane process has many advantages, including the possibility to achieve complete rejection of non-volatile components, lower operating temperatures and pressures. Furthermore, the membrane also has a compact modular construction, stationary parts, low chemical sludge effluent, absolute barriers to particles and pathogens. For example, the separation system such as water filtration that used membrane concept is more stable, and also requires small space or area. Currently, there are two types of membrane available in the market, which are polymer and ceramic types. As due to less complicated processing steps and more economical production cost with an appropriate and robustness of membrane performance therefore, polymer membranes are more favourable to be used as compared to ceramic membranes (Ahmad *et al.*, 2013).

However, due to its hydrophobic nature, polymer membrane such as polysulfone (PSf), polyethersulfone (PES), polyacrylonitrile (PAN) and polyimide (PI) membrane exhibits strong adsorption and deposition of foulants onto the

membrane surface (Rabiee *et al.*, 2015). Thus, it will reduce flux permeation and deteriorate the membrane performance.

ZnO is well known as one of the most important hydrophilic inorganic material which has been used as catalyst for chemical reactions, photo catalysts and photoelectric conversion, UV-shielding materials and especially for antibacterial agent (Li & Haneda, 2003, Ong *et al.*, 2018, Sirelkhatim *et al.*, 2015). The modification of membrane surface using ZnO as additive was successfully improved membrane performance (Rabiee *et al.*, 2015). Rabiee *et al.* (2015) reported that the addition of ZnO in polyvinyl chloride improved 88% pure water flux of membrane. This significant improvement is due to the reduction of membrane hydrophobicity. They also reported that ZnO able to enhance membrane pore size which then increased membrane flux.

According to Shen *et al.* (2012), with increased concentration of ZnO to the membrane, it decreased the contact angle value of membrane indicating the increased of hydrophilic properties. This behaviour were affected the membrane permeability as reduction of surface tension will allow more inflow of water. They also found that by addition of ZnO, thermal decomposition temperature and membrane porosity were also increased (Shen *et al.*, 2012). The multi-cycle filtration tests by Liang *et al.* (2012) showed that the modified Polyvinylidene fluoride (PVDF) with ZnO membrane demonstrated significant anti-irreversible, fouling property, suggesting that the remarkable benefit for long-term operation in practical applications. Specifically, all the membranes modified with ZnO reached almost 100% water flux recovery and were able to maintain the constant initial fluxes in multicycle filtration, whereas the raw membrane only recovered 78% and suffered continuous decline. This enhancement of recovery performance might be related to the increased of membrane surface hydrophilicity (Liang *et al.*, 2012).

Although by addition of ZnO has superior influence towards membrane properties and performance but the fact this inorganic small particles has smooth surface and easily to detach and leach out from the organic membrane structure has led to the failure of membrane operational system. Thus the improvement of additive by encapsulated with organic layer which is more compatible with the organic polymer matrix structure seems the best alternative route that can be implemented. Furthermore, the performance of bio-ZnO as an additive on polymer membrane is not yet been studied and explored. In addition, it was reported by Huang *et al.* (2013) the shape,

crystallinity and size of inorganic additives particle strongly influenced the membrane performance.

Thus in this study, the biosynthesis of ZnO was prepared by *Ananas Comosus* fruit extracts and blending with PSf membrane. The characterisation and performance of ZnO with *Ananas Comosus* fruit extracts were studied and thus compared with ZnO commercial. The influence of biosynthesis of ZnO as additive in membrane structure PSf was carried out in term of hydrophilicity, permeability, rejection of humic acid, anti-fouling and antibacterial properties of membrane. Type of water pollutant that are suitable for this research works are lake water and river as it believes to have a good antibacterial activity and also might be suitable for dye wastewater from industries as it exhibits a good photocatalytic activity.

1.2 Problem statement

Most of the membrane separation process such as food processing (Chung *et al.*, 2016), oil-water, gas separation in petroleum (Kargari & Sanaeepur, 2015) or even waste water treatment (Kargari & Sanaeepur, 2015) faced a serious problem with fouling mechanism that easily reduced membrane performance. The reduction of permeation flux due to foulants problem can be illustrated in many ways such as pore blocking, cake formation, concentration polarization and absorption.

In order to solve the fouling issues, membrane surface properties need to be improved. It was reported that hydrophilic additive will reduce membrane hydrophobicity and at the same time improve membrane performance (Leo *et al.*, 2012). The hydrophilic additives using inorganic material such as magnesium oxide (MgO) (Das *et al.*, 2018), ZnO (Agarwal *et al.*, 2017), Titanium dioxide (TiO₂) (Vishnu Kirthi *et al.*, 2011), gold (Au) (Ahmed *et al.*, 2016) and silver oxide (AgO) (Benakashani *et al.*, 2016) have been proven to improve membrane hydrophilicity and able to reduce membrane fouling resistance (Liang *et al.*, 2012). These inorganic particles are also low in cost and less hazardous.

Besides, the capability of membrane separation process is most likely depending on the medium parameter such as PH, size of foulants types of medium :the natural organic matter (NOM) that present in the permeate medium. Thus, the used of inorganic additives that not only able to improve membrane property such as

hydrophilicity property but yet able to provide photocatalytic effect that able to degrade toxic organic chemical in the membrane treatment medium. The well-known ZnO particles in degrading of methylene blue with better effect of hydrophilicity has attracted many researcher to integrate polymer with ZnO. Furthermore this inorganic material additives such as ZnO (Agarwal *et al.*, 2017) have proven able to improve the membrane characteristic and properties that has strong benefit in reducing membrane fouling. The used of these additives with various properties have claimed able to improve membrane properties such as hydrophilicity, hydrophobicity, antibacterial, self cleaning, photocatalytic and absorbance. Even most of the previous works had claimed that the used of inorganic particles able to improve membrane property and performance but the fact that these materials have different atomic and molecule structure has leads to incompatibility issue between inorganic additive and organic polymer mixed matrix membrane. Mixed matrix membrane is known as a new generation of membrane where it is a combination of polymer and inorganic filler to produce a unique properties of membrane. Theoretically, this mismatch structure and weak bonding easily break and will create the failure to membrane structure. Thus, the used of biosynthesis technique with combination fruit extract that able to encapsulated the inorganic particles has provide the best approach with simple and practical steps in reducing mismatch additive problem in polymer mixed matrix membrane or composite membrane.

As reported by Stan *et al.* (2015) biosynthesis of ZnO nanoparticles were successfully prepared using several oxidation plants extract with higher surface area, homogenous and stable condition. They reported that plants such as garlic and aloe vera assisted the growth of ZnO particles at different level with high yield (Stan *et al.*, 2015) & (Sangeetha *et al.*, 2011). As proven by most of the works these biosynthesis particles covered with organic materials have strong compatibility that led to the improvement of binding mechanism between additives particles and organic polymer mixed matrix membrane. In fact, the used of biosynthesis route not only able to produce homogenous and better characteristic of the synthesized particles that able to strengthen the mixed matrix structure, but also able to minimize the use of chemical substance. Therefore, the synthesized of ZnO nanoparticles using fruit extract of *Ananas Comosus* that has ascorbic acid as reduction agent was proposed and conducted in this work.

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