PERFORMANCE OF POLYETHYLENE GLYCOL (PEG) CAPPED ZINC OXIDE NANOPARTICLES AS PHOTOCATALYST IN MEMBRANE PHOTOCATALYTIC REACTOR FOR INDUSTRIAL DYE WASTEWATER TREATMENT

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A thesis submitted in
fulfillment of the requirement for the award of the
Degree of Master of Engineering Technology

Faculty of Engineering Technology
Universiti Tun Hussein Onn Malaysia

APRIL 2020

I dedicated this work with the deepest sense of gratitude to Almighty Allah for giving me strength, healthy life and power of mind in making my dreams come true. This work also dedicated to my beloved parents who have been my source of inspiration and continually provided their moral, spiritual and financial support in making this thesis.

ACKNOWLEDGEMENT

I thank all who in one way or another contributed in the completion of this thesis. First, I give thanks to God for protection and ability to do work. I also would like to express my deep and sincere gratitude to my research supervisor, Ts. Dr. Nur Hanis Hayati Hairom and co supervisor, Ts. Dr. Nurasyikin Misdan for giving me the opportunity to do research and providing guidance throughout this research. Their dynamism, vision, sincerity and motivation have deeply inspired me. It was a great privilege and honour to work and study under their guidance.

I must express my very profound gratitude to my parents; Desa Bin Saian and Salimah Binti Sarpan for providing me with unfailing support and continuous encouragement throughout my years of study and through the process of researching and writing this thesis. I also express my thanks to Mohamad Shafiq Aiman and my siblings for their support and valuable prayers. This accomplishment would not have been possible without them.

I thank to my friends and staffs in the Faculty of Engineering Technology for their companionship and for providing a pleasurable and friendly working atmosphere. I am truly thankful for their prompt help whenever I needed it. Lastly, I would like to thank all those people who made this thesis possible and an unforgettable experience for me.

ABSTRACT

Zinc oxide (ZnO) nanoparticles have gained immense research interest because of its high photocatalytic efficiency, inexpensive material and environmentally friendly characteristics. Several capping agents were used on ZnO nanoparticles to solve the problem concerning the agglomeration and aggregation. Nowadays, there is no study involving the effect of polyethylene glycol (PEG) as capping agent on ZnO nanoparticles via precipitation method in the application of membrane photocatalytic reactor (MPR) system. Therefore, this research focused on the effectiveness of PEG capped on ZnO as photocatalyst in MPR for treating dye wastewater from textile industry in Johor, Malaysia. Three different ZnO were utilized in the present study which are commercial ZnO, zinc oxide in the presence of polyvinylpyrrolidone (ZnO-PVP) and zinc oxide in the presence of polyethylene glycol (ZnO-PEG) nanoparticles in order to evaluate their effectiveness and effect on the membrane filtration process. Overall, it was found that ZnO-PEG nanoparticles and polypiperazine amide ultrafiltration (UF-PPA) membrane were presented as a great approach in improving normalised flux reduction (58.67%) at best operating condition and produced excellent permeate quality in terms of dye removal (100%), turbidity removal (100%), chemical oxygen demand (COD) reduction (97.37±0.06%) and total dissolved solids (TDS) removal (92.34±0.01%). The best operational conditions of MPR occurred at pH 11 of feed wastewater and 0.10g/L of ZnO-PEG loading with 6 bars of inlet pressure. The overall results were supported by Field Emission Scanning Electron Microscopy (FESEM), Atomic Force Microscopy (AFM) and contact angle analysis which confirmed that ZnO-PEG nanoparticles has great potential in improving fouling phenomenon of UF-PPA process in MPR. Moreover, it was proved that the both stages of normalised flux during the best conditions fitted well with the cake filtration model according to Wiesner and Aptel equations. Hence, ZnO-PEG nanoparticle was found to assist on the reduction of membrane fouling and achieve excellent permeate quality using MPR system for industrial dye wastewater.

ABSTRAK

Nanopartikel zink oksida (ZnO) telah mendapat minat penyelidikan yang besar kerana kecekapan fotokatalitik yang tinggi, bahan yang murah dan ciri-ciri mesra alam. Beberapa agen pelindung telah digunakan secara meluas pada nanopartikel ZnO untuk menyelesaikan masalah mengenai aglomerasi dan pengagregatan. Sehingga kini, tiada kajian yang melibatkan kesan polietilena glikol (PEG) sebagai agen pelindung pada nanopartikel ZnO melalui kaedah pemendakan diaplikasikan dalam sistem reaktor membran fotokatalitik (MPR). Oleh itu, penyelidikan ini memberi tumpuan kepada keberkesanan PEG terhadap nanopartikel ZnO sebagai fotokatalis dalam MPR untuk untuk merawat air sisa daripada industri tekstil di Johor, Malaysia.. Tiga ZnO berbeza digunakan dalam kajian ini yang merupakan nanopartikel ZnO komersial, ZnO-PVP dan ZnO-PEG untuk menilai keberkesanan dan kesannya terhadap proses penapisan membran. Secara keseluruhan, didapati bahawa nanopartikel ZnO-PEG dan membran polipiperazine amida ultra-penapisan (UF-PPA) telah mempamerkan sebagai pendekatan yang hebat dalam meningkatkan pengurangan fluks yang normal (58.67%) pada keadaan operasi yang terbaik dan menghasilkan kualiti telap yang sangat baik dari segi penyingkiran pewarna (100%), penyingkiran kekeruhan (100%), pengurangan permintaan oksigen kimia (COD) (97.37 ± 0.06%) dan jumlah pepejal terlarut (92.34±0.01%). Keadaan operasi yang terbaik MPR berlaku pada pH 11 air sisa suapan dan 0.10g/L bebanan ZnO-PEG dengan 6 bar tekanan masuk. Hasil keseluruhannya disokong oleh analisis Mikroskop Pengimbasan Pelepasan Medan (FESEM), Mikroskop Berkuatkuasa Atom (AFM) dan sentuhan sudut yang mengesahkan bahawa nanopartikel ZnO-PEG mempunyai potensi yang besar dalam memperbaiki phenomena kotoran UF-PPA proses dalam MPR. Selain itu, kedua-dua tahap fluks normal semasa keadaan yang terbaik sepadan dengan model penapisan kek menurut persamaan Wiesner dan Aptel. Oleh itu, nanopartikel ZnO-PEG telah ditemui untuk membantu pengurangan kotoran membran dan mencapai kualiti telap yang sangat baik menggunakan sistem MPR untuk air sisa pewarna industri.

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LIST OF SYMBOLS AND ABBREVIATIONS

ACs	-	Activated Carbon
AFM	-	Atomic force microscopy
AgCl	-	Silver chloride
$AgCl\text{-}SiO_2$	-	Silver chloride-doped silica
Al	-	Aluminium
AnMBR	-	Anaerobic Membrane Bioreactors
BET	-	Brunauer-Emmett-Teller
BOD	-	Biochemical oxygen demand
CdS	-	Cadmium sulphide
CdSe	-	Cadmium selenite
CO_2	-	Carbon dioxide
COD	-	Chemical Oxygen Demand
Cr (VI)	-	Hexavalent chromium
CuO	-	Copper oxide
DoE	-	Department of Environment
EQA	-	Environment Quality Act
Fe	-	Iron
Fe_2O_3	-	Iron(III) oxide
FESEM	-	Field-Emission Scanning-Electron Microscopy
GaAs	-	Gallium arsenide
GaP	-	Gallium phosphide
GS	-	Gas separation
H_2O	-	Water
HA	-	Humic acid
HAA	-	Halo acetic acids
HCl	-	Hydrochloric acid
IEP	-	Isoelectric point
IUPAC	-	International Union of Pure and Applied Chemistry

K	-	Fitted parameter constant
MF	-	Microfiltration
MPR	-	Membrane photocatalytic reactor
MWCO	-	Molecular weight cut-off
Na_2SO_4	-	Sodium sulphate
NaOH	-	Sodium hydroxide
NF	-	Nanofiltration
NOM	-	Natural organic matters
O_2^-	-	Superoxide anion radicals
OH^-	-	Hydroxide ion
OH·	-	Hydroxyl radicals
PEG	-	Polyethylene glycol
PES	-	Polyethersulfone
POMSE	-	Palm oil mills secondary effluents
PP	-	Polypropylene
PPA	-	Polypiperazine amide
PMMA	-	Poly (methyl methacrylate)
Pt	-	Platinum
PTFE	-	Polytetrafluoroethylene
PVA	-	Polyvinyl alcohol
PVDF	-	Polyvinylidene fluoride
PVP	-	Polyvinylpyrrolidone
R^2	-	Linear line coefficient
RB19	-	Reactive Blue 19
RMS	-	Root mean square
RO	-	Reverse osmosis
Ru	-	Ruthenium
SiC	-	Silicon carbide
SiO_2	-	Silicon oxide
SnO_2	-	Tin oxide
$SrTiO_3$	-	Strontium titanate
TDS	-	Total dissolved solids
TEM	-	Transmission electron microscopy
TiO_2	-	Titanium dioxide

TMP	-	Trans-membrane pressure
TOC	-	Total organic carbons
TSS	-	Total suspended solids
UF	-	Ultrafiltration
UV	-	Ultraviolet
VOCs	-	Volatile organic compounds
WO_3	-	Tungsten (VI) oxide
XRD	-	X-ray diffractometer
ZnO	-	Zinc oxide
ZnO-PEG	-	Zinc oxide in the presence of polyethylene glycol
ZnO-PVP	-	Zinc oxide in the presence of polyvinylpyrrolidone
ZnO-PVP-St	-	Zinc oxide under stirring in presence of polyvinylpyrrolidone
ZnS	-	Zinc sulphide
ZrO_2	-	Zirconium oxide

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CHAPTER 1

INTRODUCTION

1.1 Research Background

In the present scenario, textile industry is often associated with the environmental issue due to consumption of large amounts of water and variety of chemical wasted during dyeing and finishing processes (Paraschiv, Tudor & Petrariu 2015) (Choudhary & Islam, 2017). Generally, the effluent from textile industry is often rich in colour, residues containing chemicals and reactive dye (Sowmyashree *et al.,* 2015). In-depth, the negative environmental effects can include aesthetic problems, obstruction of sunlight and interference with aquatic biological processes that modifies the balance of the ecosystem (Wijannarong *et al.,* 2013). As reported by Hairom and co-workers, the discharge of dye wastewater constitutes a threat to the environment and human health. Furthermore, the releasing untreated wastewater from textile industry are known to be one of most difficult to treat due to the recalcitrant nature of dye (Rashidi, Sulaiman, & Hashim, 2012). The presence of dye even at very low concentrations could highly be visible and undesirable. The dye effluents generally can be characterised by high chemical oxygen demand (COD), pH, turbidity, and colour (Alcaina-Miranda *et al.,* 2009). Yao and co-workers reported that the amounts of COD and total dissolved solids (TDS) remained in the textile wastewater although more than 90% of pollutants has been removed by using the biochemical processes (Yao *et al.,* 2016). Indeed, dye are stable to light and heat due to their complex aromatic structures (Rizzi *et al.,* 2017). Thus, in order to limit the environmental impact, the textile wastewater must be adequately treated before being discharged into the receiving water bodies.

Currently, conventional wastewater treatment methods based on physical, biological or chemical processes were typically anticipated to protect the public from exposure to organic dye and their derivatives (Su *et al.,* 2016) (Komolafe *et al.,* 2013). These approaches are undesirable method since not fully degraded and its ability to convert into another pollutant. Many researchers agreed that adsorption method as the alternative in the environment treatment system application (Zhang *et al.,* 2015) (Rashed, 2013). However, this treatment consists of complex processes, time-consuming, usage of hazardous reagents and requirement of specific equipment. Thus, photocatalysis is one of the promising method for exhibit higher efficiency in the degradation of dye wastewater and disinfection of pathogenic microorganisms (Zheng *et al.,* 2017). In spite of these advantages, the main limitation for photocatalytic activity is the separation of photocatalyst in slurries from the treated wastewater for reusability purpose. Alternatively, the use of a membrane photocatalytic reactor (MPR) could potentially achieve more sustainable operations (Mozia, 2010). Typically, the MPR ensures efficient degradation of dye, which have gained considerable attention in wastewater treatment in recent years. The MPR is a hybrid system demonstrated by the coupling of photocatalysis process and membrane filtration system (Hairom *et al.,* 2014). It has attracted extensive attention to researchers as a potential dye wastewater treatment to compete with existing treatment in the future. It also shows remarkable improvement over the existing treatment and has potential impacts for the industrial dye wastewater treatment. This is due to several advantages such as low consumption of energy, not require large area for installation and continuous process (Hairom *et al.,* 2014a).

Photocatalyst played a critical role that influenced the MPR performance in producing a better quality of treated wastewater (Zheng *et al.,* 2017). Recent studies reported that zinc oxide (ZnO) nanoparticle has well known as effective photocatalyst due to their reasonable properties which are strong excitation binding energy, higher reactivity, surface area, photosensitivity, non-toxic nature and chemical stability (Hairom *et al.,* 2014a & 2015) (Sudha & Rajarajan, 2013) (Bhatia & Verma, 2017) (Kayani, Saleemi, & Batool, 2015). In addition., ZnO has been widely used due to the stable wurtzite structure and wide band gap (3.2eV) that suitable as a photocatalyst for dye wastewater treatment (Hairom *et al.,* 2015). ZnO nanoparticles are commercialised for various applications such as sensor (Lu *et al.,* 2012) (Lacy *et al.,* 2008), solar cells (Hau *et al.,* 2008), light emitting diode (Lee *et al.,* 2010) and photocatalysis (Bhatia & Verma, 2017). However, the ZnO nanoparticles have the tendency to agglomerate in which resulting from the Ostwald ripening and Van der Waals interactions between nanoparticles. The previous study also reported that ZnO nanoparticles are aggregated severely due to the high surface energy of ZnO nanoparticles (Raoufi & Raou, 2013). Therefore, these problems generally resolved by the capping technique of ZnO nanoparticles in which predicted to controls the particles size, reduces the agglomeration and obtain well-dispersed nanoparticles.

Membranes technologies provide an important solution in environmental fields such as recycling valuable components from the waste streams, pollution reduction and water reuse. There are two important parameters that affect the efficiency of membrane processes which are rejection and permeate flux (Koyuncu, Topacik, & Wiesner, 2004). Lately, the utilization of membrane separation process is growing fast due to a more restrictive legislation concerning industrial wastewater and the necessity of water resources. According to Aouni *et al.*, (2012), they reported that the membrane techniques have potential to remove the dye stuffs, allow the reuse of the auxiliary chemicals used for dyeing, concentrate the dyestuffs and auxiliaries and produce purified water. There have been many investigations on the treatment of the dye wastewater using microfiltration (MF) (Gopakumar *et al.*, 2017) (Saini, Bulasara, & Reddy, 2018), ultrafiltration (UF) (Lin *et al.*, 2016) (Alventosa-Delara *et al.*, 2014), and nanofiltration (NF) membranes (Buscio *et al.*, 2016). A special attention was given to the NF membrane that owing to its well-balanced performance between water flux and solute rejection as well as relatively low operations. UF and MF are inherently constrained by membrane fouling with dissolved organic substances such as humic acid (HA) and proteins which acting as major foulants. The previous study reported that lower quality of permeate during the both MF and UF membranes since small particles, molecules and ions can pass easily through the membranes (Hairom, Mohammad, & Kadhum, 2014a). On the other hands, the accumulation of species at the membrane surface adversely affects the process performance since it leads to an increase in the membrane resistance over time. Hence, a primary treatment is required for the further improvement in both flux and separation efficiency during the filtration of textile wastewater. The efficiency of the hybrid MPR system in the presence of polymer capped ZnO was investigated in mitigating membrane fouling and analysing the effect of permeate quality. The outcomes of this research can provide instructive information on the best operational conditions during the MPR treatment.

1.2 Problem Statement

The greatest technical challenges during the synthesis of ZnO nanoparticles are agglomeration and aggregation. There has been a lot study reported that the uncapped ZnO has no particular morphology and the particles are highly agglomerated (Chandrasekaran, Viruthagiri, & Srinivasan, 2012). In addition, the particle size obtained by uncapped ZnO is much bigger than capped ZnO since the capping agent was used to control the growth by formed a shell surrounding the particles (Yogamalar, Srinivasan, & Bose, 2009). The previous study by Saravanan and co-workers investigated the formation of cadmium sulphide (CdS) nanoparticles capped by PVP (polyvinylpyrrolidone) were synthesised via chemical co-precipitation method. It can be inferred that PVP can lower the surface energy of the nanoparticles, and hence the PVP capped CdS nanoparticles shows excellent monodispersed property (Saravanan *et al.*, 2011). Generally, PVP tends to modify the Ostwald ripening kinetics in such a way that the growth rate decreases with the size of the CdS nanoparticles and effectively narrow the size distribution. Furthermore, the previous study reported that capping agent of PVP has ability to reduce the formation of agglomeration and aggregation of ZnO nanoparticles (Hairom *et al.*, 2014). They also reported that ZnO-PVP nanoparticles was the best photocatalyst in hybrid treatment system of MPR for dye wastewater treatment that exhibited the highest photodegradation efficiency. However, ZnO-PVP nanoparticles was resulting in severe flux decline and membrane fouling.

The choice of capping agents has placed much emphasis since the properties which included size, shape and the interaction with solvent surroundings of the nanoparticle are strongly influenced to the synthesised ZnO nanoparticles (Toh, Jurkschat, & Compton, 2015). The interesting properties of PEG have been studied well in the biotechnology and medicine field for controlled drug released compounds since PEG are inexpensive, stable under ambient condition and do not release volatile organic compounds (VOCs). Furthermore, many researchers have reported preparation of noble metal nanoparticles using PEG, exploiting its non-toxic, non-irritating and moisturizing properties (Sudha *et al.*, 2013). PEG is known to have long hydrocarbon chain structures with hydrophobic ends. The number of monomers in PEG is critical in manipulating the nanoparticle sizes. The rate of particle aggregation is a major factor that controls the morphology and crystallinity of the final product. Besides that, it has been reported that PEG with uniform and ordered chain structure is easily absorbed at the surface of metal oxide colloid (Thirugnanam, 2013). Based on previous study, PEG was used as a templating reagent for producing synthesized microstructure titanium dioxides (TiO_2) photocatalyst via sol–gel method (Sun *et al.*, 2008). PEG molecules acted significantly in well-controlled crystal growth and the enlargement of surface area via the pore-forming function. Although there has been an increasing amount of studies on polymer capped on ZnO, the effectiveness of PEG capped on ZnO nanoparticles to improve MPR system has not been discovered yet in the previous study. Therefore, these details are very useful to improve the photocatalyst performances in the photocatalytic activity and membrane filtration process. The present study attempted to obtain the best conditions of MPR by studying the effect of different capping agents on ZnO nanoparticles.

1.3 Objectives of the Study

The objectives of this research are:

(i) To study the effectiveness of different ZnO photocatalyst in membrane photocatalytic reactor (MPR) for industrial dye wastewater treatment.

(ii) To determine the flux decline, membrane fouling and permeate quality after MPR process regarding to the selected capped ZnO and membranes types, and influence of operating parameters for the MPR system.

(iii) To study the mechanisms of membrane fouling using model fitting according to the theory of blocking filtration laws.

1.4 Scopes of the Study

The scopes of this research are:

(i) Study the self-synthesised ZnO capped by the different types of polymers (PVP and PEG) via precipitation method by using Transmission Electron Microscopy (TEM), X-ray diffractometer (XRD) and Brunauer-Emmett-Teller (BET), respectively.

(ii) Study the permeability performance on the different types of membranes (NF-PPA, UF-PPA, MF-PES and MF-PVDF) under different pressure (3, 4, 5 and 6 bars) in the MPR system.

(iii) Study the effectiveness of photocatalyst (ZnO-PEG, ZnO-PVP, commercial ZnO and absence ZnO) in MPR for industrial dye wastewater treatment under different types of membrane (NF-PPA, UF-PPA, MF-PES and MF-PVDF), initial pH (pH 4-13), ZnO loading (0.08-0.30 g/L), dilution of dye wastewater (0-75% dilution) and pressure (4-7bars).

(iv) Determination of the flux decline and membrane fouling in terms of membrane surface and cross-section morphology by using Field Emission Scanning Electron Microscopy (FESEM), surface roughness analysis by using Atomic Force Microscopy (AFM) and contact angle based on standard sessile drop method.

(v) Determination of the permeate quality in terms of colour intensity, turbidity, and chemical oxygen demand (COD) and Total Dissolved Solids (TDS).

(vi) Study for the membrane fouling mechanism during the best conditions using model fitting according the theory of blocking filtration laws that have been modified by Wiesner & Aptel, (1996).

1.5 Significance of the Study

The findings of this study will redound to the benefits of MPR in the long term for treating dye wastewater by maximizing degradation efficiency of dye wastewater and minimizing membrane fouling to enhance the technology of wastewater treatment plant. The greater demands in textile industries justified the need for more effective in treatment approaches. Thus, the present study applied the recommended approach of treatment in which significance on technical and economical to the industry that produced effluent based on dye wastewater. It is beneficial to save energy as well as to cut down the size of installation due to additional operations such as coagulation, flocculation and sedimentation are not necessary for this reactor.

Moreover, ZnO-PEG nanoparticle via precipitation method provide significant boost to the development of synthesised ZnO since it contributed to the manufacturer of ZnO nanoparticles. The improvement on synthesised ZnO influenced to the performance of MPR system. Furthermore, this is a preliminary study of MPR before it could be applied in the pilot scale of wastewater treatment plant project. Therefore, the present study provided an elucidation of the effectiveness of different capping agents on ZnO nanoparticles in MPR, behavior of the used membranes and membrane fouling mechanisms in order to develop advanced treatment systems that have potential to meet the stringent imply of effluent discharge regulations.

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