PRODUCTION, CHARACTERIZATION AND UTILIZATION OF EPOXY SPHERICAL MEMBRANE FILTER PREPARED BY AN ADVANCED AQUEOUS METHOD

SEA BEE ING

UNIVERSITI MALAYSIA PERLIS

2014

Production, Characterization and Utilization of Epoxy Spherical Membrane Filter prepared by an Advanced Aqueous Method

by

SEA BEE ING
1330410924

A thesis submitted in fulfillment of the requirements for the degree of Master of Science in Materials Engineering

School of Materials Engineering
UNIVERSITI MALAYSIA PERLIS

2014

ACKNOWLEDGEMENT

First and foremore, I feel grateful to God as a creator of our universal had provided us with plentiful and valuable natural resource and wonderful environment. God at the same time had gave me strength to complete my master research which is one of the toughest challenges in completing my degree. I would like to express my deepest gratitude to my project supervisor Dr. Du Ngoc Uy Lan for the opportunities he had gave me to learn and to complete the project under his supervision. He had been a great mentor and always opened to discussion with useful suggestions and advice. His support and guidance along the project, and an opportunity for me to progress in this project had been invaluable. I am very grateful as he shared his wealth of knowledge in polymer field especially at the field of epoxy with me.

Special thanks to Dr. Ong Hui Lin, my co-supervisor for her comments and suggestions to the project. Besides, her initiative to involve herself to the project by held group discussion on related journal. She had shared her knowledge and discussion had been carried out in this discipline during our discussion. There are many who had contributed directly or indirectly helped me in completing my studies. I would like to take this opportunity in devoting deepest thanks to all School of Material Engineering staffs who had lent their hand in my experiments. I also would like to acknowledge Ministry of Higher Education Malaysia for providing me FRGS grant (grant no: 9001-00326) that supported this project.

This study could not be realized without unconditional love and support from my family. Even though they were miles away, they were always with me. Their encouragement and concern had motivated me to carry on during this study. I would like to extend my grateful appreciation and thanks to my colleague and friends for their friendship and support and all postgraduate students of Materials Engineering, UniMAP are also acknowledge.

TABLE OF CONTENTS

CHAPTER 3 RESEARCH METHODOLOGY

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

10E:6PA	10 Epoxy: 6 Polyamide
10E:10PA	10 Epoxy: 10 Polyamide
3R:7W	3 Resin: 7 Water
4R:6W	4 Resin: 6 Water
5R:5W	5 Resin: 5 Water
API	American Petroleum Institute
ASTM	American Standard for testing and Materials
ATRP	Atomic transfer radical polymerization
BLD	Blue dextran
BSI	British standard institute
CA	Cellulose acetate
$CaCO_3$	Calcium carbonate
C_p	Specific heat capacity
DGEBA	Diglycidyl ether of bisphenol A
DSC	Different scanning calorimetric
DTGA	Derivative thermal gravimetric analysis
E	Epoxy
ECMR	Electrocatalytic membrane reactor
EO	Engine oil solution
ESM	Epoxy spherical membrane
ESMC	Epoxy spherical membrane column

ESMC-0	Epoxy spherical membrane column without calcium carbonate
ESMC-25	Epoxy spherical membrane column filled with 25phr calcium carbonate
ESMC-50	Epoxy spherical membrane column filled with 50phr calcium carbonate
FTIR	Fourier Transform Infrared Spectroscopy
GCC	Ground calcium carbonate
GMA	Glycol methacrylate
HFP	Hexafluoropropylene
HIPE	High internal phase emulsion
IMS	Integrated membrane systems
LRP	Living radical polymerization
LT	Low temperature
MBR	Bioreactor membrane
MF	Microfiltratiom
NIPS	Non-solvent induced phase separation
O	oil
O/W	Oil-in-water
O/W/O	Oil-in-water-in oil
OSN	Organic solvent nanofiltration
PA	Polyamide
PACM	1, 4,40-methylenebiscyclohexanamine
PAN	Polyacrylonitrile
PCC	Precipitated calcium carbonate

PDMS	Polydimethylsiloxane
PE	Polyethylene
PEG	Poly (Ethylene Glycol)
PES	Polyethersulfone
PHEMA	Poly(2- hydroxyethyl methacrylate)
PIPS	Polymerization induced phase separation
PIs	Polyimides
PLA	*Poly(Lactic acid*)
PLGA	Poly(Lactic acid-co-Glycolic acid)
PLGA	Poly (lactide-co-glycolide)
PLLA	Poly(L-lactic acid)
PMMA	Poly(methyl methacrylate)
PP	Polypropylene
PS	Polystyrene
PSU	Polysulfone
PO	Palm oil solution
PVDF	Polyvinylidene fluoride
R	Resin
RBD	Refined, bleached and deodorized
RT	Room temperature
SAE	Society of Automotive Engineers
SEM	Scanning electron microscopy
T_g	Glass transition temperature
TGA	Themalgravimetric analysis

THF	Tetrahydrofuran
TIPS	Thermally induced phase separation
UF	Ultrafiltration
UV	Ultraviolet
W	Water
W/O	Water-in-oil
W/O/W	Water-in-oil-in-water

LIST OF SYMBOLS

%	Percentage
℃	Celcius
Al	Aluminium
Al_2O_3	Aluminium oxide
$CaCO_3$	Calcium carbonate
Ca^{2+}	Calcium ion
cm^3	Centimeter cubic
eq	Equivalent
eg	Example
g	Gram
g/ml	Gram/ Mililitre
hrs	Hours
J	Joule
kg	Kilogram
kN	Kilo Newton
kV	Kilo Volt
$MgCl_2$	Magnesium chloride
$MgSO_4$	Magnesium sulfate
mM	Mili Molar
mm	Mili Meter
mmol	Mili Moles

Pa	Pascal
s	Second
N	Newton
NaCl	Sodium Chloride
nm	Nanometer
pH	Potential Hydrogen
phr	Parts Per Hundred
ppm	Parts Per Million
rpm	Rotation Per Minute
TiO_2	Titanium Oxide
wt.	Weight
vol.	Volume
Zn	Zinc
ZrO_2	Zirconium Dioxide
μm	Micrometer

LIST OF EQUATIONS

Penghasilan, Pencirian dan Penggunaan Penapis Membran Sfera Epoksi disediakan dengan Kaedah Akueus Termaju

ABSTRAK

Zaman sekarang, masyarakat mula mempersoalkan keselamatan air. Bahan pencemar berbahaya dalam air dapat mengancam nyawa hidupan dan ia tidak boleh diabaikan Walaupun banyak produk yang canggih telah dicipta digunakan dalam terapi air sisa, tetapi kos pengendalian membran yang tinggi dan kekurangan bekalan mengehadkan perkembangan aplikasi tersebut. Dengan itu, idea baru untuk membran sfera epoksi (ESM) yang mempunyai liang berskala mikro dihasil untuk industri rawatan air sisa. Dalam kajian tersebut, kaedah akueus lanjutan digunakan untuk menghasilkan ESM berdasarkan teknik emulsi air di dalam minyak dalam air (W/O/W). Kaedah ini mudah dan mesra alam kerana tiada pelarut digunakan dan tiada pelepasan produk yang meruap. ESM sesuai dihasilkan dalam kuatiti yang besar. Epoksi dan poliamida berada dalam bentuk cecair bawah suhu bilik membolehkan emilsifikasi berlaku. Poliamida yang reaktif berasal daripada minyak sayur dipilih di mana ia bertemu dengan semua syarat untuk membolehkan proses emulsifikasi tanpa kehadiran surfaktan dan pengemulsi. Kalsium karbonat diisi dalam ESM sebagai penggalak pengemulsi untuk menghasilkan zarah epoksi yang halus. Ia dapat meningkatkan sifat-sifat mekanikal, kestabilan terma, keupayaan koagulan minyak semasa penapisan dan dapat kurangkan kos pembuatan. Air suling bertindak sebagai fasa tersebar dalam titisan epoksi dan fasa berterusan dalam sistem emulsi. Air suling sebagai template dalam ESM dan mengewap mewujudkan liang yang kompleks dalam ESM ketika mengawetan. Membran sfera epoksi turus (ESMC) dibina daripada zarah-zarah epoksi dengan struktur liang yang kompleks membolehkan prinsip membran sfera diaplikasi untuk penapisan dan pemisahan. ESMC didapati berkesan dalam penapisan karbon hitam dan serbuk tembaga dengan kadar aliran efluen yang rendah. Pemisahan minyak kelapa sawit dengan air dan minyak enjin dengan air berjaya dipisahkan dengan daya graviti di mana air jernih mengalir keluar dahulu kemudian diikuti dengan minyak. Ruang turas kaca mempersembahkan kecekapan penapisan yang lebih baik daripada ruang turas PP. Diameter ruang turus yang lebih besar membenarkan kadar aliran yang pantas tetapi kekurangan keberkesanan penapisan.

Production, Characterization and Utilization of Epoxy Spherical Membrane Filter prepared by an Advanced Aqueous Method

ABSTRACT

In recent years, the public raise important questions about the safety of water. The threat of harmful contaminants in water can no longer be ignore. Although many advanced products have been develop and applied in wastewater treatment, however these membranes are expensive in operating cost and short supply prohibiting their wide application. Thereby, the idea to produce an epoxy spherical membrane (ESM) with multiple micro-scale pores for industry wastewater treatment is developed. In this research, an advanced aqueous method is use to produce ESM based on the water in oil in water (W/O/W) double emulsion polymerization technique. This method is simple and environmentally friendly because no solvent is used; no emission of volatile product. It is feasible to scale up. Epoxy and polyamide are in liquid form at room temperature that enables to be emulsified. Reactive polyamide originated from vegetable oil was selected, which has met all the requirements to be able emulsifying without surfactants nor emulsifier. Calcium carbonate filled in ESM acted as an emulsifying promoter producing fine epoxy particles. It also improved the mechanical properties, thermal stability, and oils coagulation ability during filtering and reduced the manufacturing cost. Distilled water acted as a dispersed phase in epoxy droplets and continuous phase in emulsion system. It worked as a template in ESM and vaporized during curing creating a complex pore structure in ESM. ESMC (epoxy spherical membrane column) built from epoxy particles with complex pore structure enabled to apply the spherical membrane principle for filtering and separation. ESMC was founds effective in removing the contaminated carbon black and copper powder from the water but allow slower effluent flow rate. Separation behavior of palm oil-water and engine oil-water solutions were successfully gravitationally separates whereby clear water was flow out first then followed by oils. Glass column was perform a better filtering efficiency than PP column. Larger diameter of column allowed faster filtrate's flow rate but poorer filtering effectiveness.