

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

SEA BEE ING

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UNIVERSITI MALAYSIA PERLIS

2014



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

by

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A thesis submitted in fulfillment of the requirements for the degree of
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**School of Materials Engineering
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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 | Microfiltration |
| 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 | <i>Poly(Lactic acid)</i> |
| 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 | Thermalgravimetric 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 |

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LIST OF SYMBOLS

| | |
|--------------------------------|--------------------|
| % | Percentage |
| °C | Celcius |
| Al | Aluminium |
| Al ₂ O 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 sulfete |
| 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 |

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LIST OF EQUATIONS

| NO. | | PAGE |
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| 2.1 | Porosity | 29 |
| 3.1 | Bulk density | 46 |
| 3.2 | Filtrate's flow rate | 49 |
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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 kuantiti yang besar. Epoksi dan poliamida berada dalam bentuk cecair bawah suhu bilik membolehkan emulsifikasi 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.