THE APPLICATION OF BLENDED SURFACTANT IONIC LIQUID IN EMULSION LIQUID MEMBRANE FOR PHENOL REMOVAL FROM AQUEOUS WASTE SOLUTION

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To my beloved parent, siblings, relatives, supervisor and all my friends.

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ABSTRACT

Emulsion liquid membrane (ELM) is a liquid membrane technology that has the capability to remove metals and organic compounds from aqueous waste solution in high efficiency. Phenol as one of organic compounds that is toxic and has carcinogenic effect. Phenol exists in industrial effluent especially from petrochemical, paint, pharmaceutical industries, and refineries. However, application of ELM in industries is still limited due to the instability of emulsion membrane during the process. Thus, this research investigates the significant use of blended surfactant in emulsion preparation and the effect of ionic liquid on emulsion stability and extraction performance of ELM process. Selection of liquid membrane components was carried out to determine the optimum ratio of palm oil to kerosene as a solvent and to determine the suitable stripping agent to be used. In this research, 7:3 ratio of palm oil to kerosene was selected as the liquid membrane phase and sodium hydroxide as the stripping agent phase. Then, blended surfactant emulsion liquid membrane was formulated using several composition of mixed surfactant of Span 80 and Tween 80. The stability of primary water-in oil (W/O) emulsion was studied by manipulating several parameters such as hydrophile-lipophile balance (HLB) range, blended surfactant concentration, homogenizer speed, emulsifying time, and organic membrane to internal (O/I) ratio using one factor at one-time method. The results showed that the most stable emulsion was obtained at HLB 5, 3% (w/v) of blended surfactant concentration, 8000 rpm homogenizer speed, 3 minutes emulsifying time and 3:1 O/I ratio. Using this stable W/O emulsion, the extraction performance of phenol was studied by manipulating several parameters such as agitation speed, extraction time, ionic liquid concentration and treat ratio (emulsion phase to feed phase ratio). This system was optimized using the response surface methodology. The results showed that the optimum conditions were obtained at 267 rpm agitation speed, 5 minutes extraction time, 0.107% (w/v) ionic liquid concentration and 1:7.3 treat ratio where 83% of phenol was extracted. At this condition, the recovered phenol obtained was about 11 times enrichment ratio as concentrated phenolate solution. In conclusion, blended surfactant ionic liquid has the potential to increase emulsion stability, phenol extraction and recovery.

ABSTRAK

Membran cecair emulsi (ELM) merupakan teknologi membran cecair yang mempunyai keupayaan untuk menyingkirkan logam dan sebatian organik daripada air sisa buangan pada kecekapan yang tinggi. Fenol adalah salah satu sebatian organik yang beracun dan mempunyai kesan karsinogenik. Fenol wujud dalam sisa buangan industri terutamanya daripada industri petrokimia, cat, farmaseutikal, dan loji penapisan. Bagaimanapun, penggunaan ELM dalam industri masih terbatas disebabkan ketidakstabilan membrane emulsi semasa proses tersebut. Oleh itu, kajian ini menyiasat kesan signifikan penggunaan adunan surfaktan dalam penyediaan emulsi dan kesan cecair ionik terhadap kestabilan emulsi dan prestasi pengekstrakan di dalam proses ELM. Pemilihan komponen membran cecair telah dijalankan untuk menentukan nisbah optimum campuran minyak sawit dan kerosin sebagai pelarut dan untuk menentukan agen pelucutan yang sesuai digunakan. Kajian ini, nisbah 7:3 campuran minyak sawit dan kerosin dipilih sebagai membran cecair dan natrium hidroksida sebagai agen pelucutan. Seterusnya, membran cecair emulsi surfaktan teradun dirumus dengan menggunakan beberapa komposisi campuran surfaktan Span 80 dan Tween 80. Kestabilan emulsi air-dalam-minyak (W/O) telah dikaji dengan memanipulasi beberapa parameter seperti nilai keseimbangan julat hidrofilik-lipofilik (HLB), kepekatan surfaktan teradun, kelajuan penghomogen, masa pengemulsian, dan nisbah membran organik kepada fasa dalaman (O/I) menggunakan kaedah satu faktor pada satu masa. Hasil menunjukkan bahawa emulsi yang paling stabil diperolehi pada HLB 5, kepekatan surfaktan teradun 3% (w/v), kelajuan penghomogenan 8000 rpm, masa pengemulsian 3 minit dan nisbah O/I 3:1. Dengan menggunakan emulsi W/O yang stabil, prestasi pengekstrakan fenol dikaji dengan memanipulasi beberapa parameter seperti kelajuan pengaduk, masa pengekstrakan, kepekatan cecair ionik dan nisbah rawatan (nisbah fasa emulsi kepada fasa suapan). Sistem ini dioptimumkan dengan menggunakan kaedah tindakbalas permukaan. Hasil kajian menunjukkan bahawa keadaan optimum dicapai pada kelajuan pengaduk 267 rpm, masa pengekstrakan 5 minit, kepekatan cecair ionik 0.107%(w/v) dan nisbah rawatan 1:7.3 di mana 83% daripada fenol berjaya diekstrak. Pada keadaan ini, perolehan semula fenol sebagai larutan fenolat pekat adalah pada 11 kali nisbah pengayaan. Kesimpulannya, cecair ionic surfaktan teradun berpotensi untuk meningkatkan kestabilan emulsi, pengekstrakan dan penghasilan semula fenol.

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LIST OF SYMBOLS

w	-	Weight
g	-	Gram
mg	-	Milligram
v	-	Volume
L	-	Litre
mL	-	Millilitre
m	-	Meter
cm	-	Centimeter
nm	-	Nanometer
M	-	Molar
ppm	-	Part per million
hr	-	Hour
min	-	Minute
s	-	Second
rpm	-	rotation per minute
˚C	-	Degree Celsius
%	-	Percent

LIST OF ABBREVIATIONS

BLM	-	Bulk liquid membrane
ELM	-	Emulsion liquid membrane
SLM	-	Supported liquid membrane
HLM	-	Hybrid liquid membrane
LM	-	Liquid membrane
ppm	-	Part per million
rpm	-	Rotation per minute
min	-	Minutes
BPA	-	Bisphenol A
CDs	-	Compact disks
MSDS	-	Material Safety Data Sheet
LLE	-	Liquid-liquid extraction
TBP	-	Trybutyl phosphate
NaOH	-	Sodium hydroxide
ACs	-	Activated carbon
GACs	-	Granular activated carbon
PACs	-	Powdered activated carbon
BFA	-	Bagasse fly ass
ACC	-	Activated carbon-commercial
ACL	-	Activated carbon-laboratory
ZIF-67	-	Zinc-methylimidazolate framework-67
ZIFs	-	Zeolitic imidazolate frameworks

MEUF	-	Micellar-enhanced ultrafiltration
FSSLM	-	Flat sheet supported liquid membrane
HFLM	-	Hollow-fiber liquid membrane
FLM	-	Flowing liquid membrane
MHS	-	Multi-membrane hybrid membrane
BLLS/RBL	-	Bilinear least squares/residual bilinearization
AR	-	Allura red
SY	-	Sunset yellow
HFSLM	-	Hollow fiber supported liquid membrane
FSSLM	-	Flat sheet supported liquid membrane
SBR	-	Styrene Butadiene Rubber
PIB	-	Polyisobutylene
HLB	-	Hydrophile-lipophile balance
RTILs	-	Room temperature ionic liquids
ILMC	-	Ionic Liquid Mixed Carrier
CCD	-	Central Composite Design
RSM	-	Response surface methodology
Span 80	-	Sorbitan monooleate
Tween 80	-	Polysorbate 80
ANOVA	-	Analysis of variance
DOE	-	Design of experiment
BBD	-	Box-Behnken Design
W/O	-	Water in oil
W/O/W	-	Water in oil in water
O/W/O	-	Oil in water in oil

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CHAPTER 1

INTRODUCTION

1.1 Research Background

In recent years, the production of organic compounds in wastewater increases significantly every hour and could become one of the major problems to the environment if not properly discharged. Despite of the useful application of the chemical, the disposal or emission of it could bring negative impact to the environment. The increase of organic waste in water body will affect aquatic organisms as well as human health. In order to overcome the problem, separation and removal of organic compounds from industrial waste effluents offer a good alternative.

In the past years, a lot of study on separating and recovering organic compounds from wastewater were established and commercialized such as distillation, adsorption, solvent extraction, ultrafiltration process and membrane separation. On top of that, it is reported that the liquid membrane technology is a promising method to extract and recover organic compound. Liquid membrane technology is a simple operation that promotes high separation selectivity as well as high efficiency on extraction of inorganic and organic compound with low process costs (Kaminski and Kwapinski, 2000).

Liquid membrane technology is widely used in biotechnology, food and pharmaceutical industries (Roman *et al.*, 2010). Liquid membrane technology is a system which separates two aqueous phase by selective permeation of solutes through an organic barrier. The process of mass transfer can be explained in three liquid phases which are feed phase, membrane phase and receiving phase and can be carried out in bulk liquid membrane (BLM), supported liquid membrane (SLM) and emulsion liquid membrane (ELM) processes. Unfortunately, the important usage of liquid membranes technology are currently inadequately high. The loss of the membrane solvent and carrier are the main issue for all types of supported liquid membranes. Meanwhile in emulsion liquid membrane process, the main disadvantage of the process is the emulsion instability. Therefore, the development of process design and new formulation of liquid membrane need to be investigated to improve the performance and efficiency of the process.

ELM is one of the recent and promising technology that has high potential in separating metals and organics from wastewater. ELM process is consists of three steps such as emulsification, extraction and demulsification. The main advantages of ELM include lower solvent and carrier consumption than in the case of conventional extraction and provide very large interfacial area that affect the process kinetics. In addition, ELM also offers attractive advantages such as simple operations, large surface contact area, high efficiency and low operational expenses (Othman *et al.*, 2017).

Therefore, with this advantages, the extraction of phenol was investigated. Phenol is considered as pollutant to the environment due to its toxicity and carcinogenic effect. Therefore, treatment of wastewater containing phenol before being discharge has become the recent major concern. In contrast, phenolic compounds have been used as adhesives, dyes, germicides and chemical intermediate and are considered as valuable chemical products (Othman *et al.*, 2015). Today, an assortment of creative techniques for treatment of phenolic compounds is developed to remove the undesirable phenol from wastewater.

Thus, this research study and optimize the stability of ELM process using blended surfactant as the notion to improve the stability of the emulsion in phenol extraction. Several factors and parameters that affects the membrane stability and extraction of phenol were investigated to identify the optimum conditions of the process. The extraction of phenol was designed and optimized using the response surface methodology (RSM).

1.2 Problem Statement

Lately, it is accounted for that liquid membrane technology has extraordinary potential in removing heavy metals and major organic pollutants from industrial wastewater. ELM is a simple operation that offers many advantages but unfortunately, ELM is not fully commercialized due to some limitation. Thus, a lot of study was done in order to make ELM more applicable in the industries.

Phenol is a major pollutant in wastewater due to its presence in the effluent of major processing and refining plants, which could cause severe effects if not treated carefully. There are various techniques of phenol removal and recovery such as adsorption, liquid–liquid extraction (LLE), steam distillation, chemical oxidation and biodegradation, while advanced techniques incorporate photo oxidation processes and membrane separation technologies (Mohammadi *et al.*, 2015). However, each method has its own limitations and disadvantageous throughout the process. Therefore, the use of ELM in order to remove phenol from wastewater has been chosen as one of the treatment alternative. The phenol removal from aqueous solution could be increase and optimize by investigating the operating process parameters of ELM process.

The major limitation of ELM is the instability of the emulsion such as swelling and breakage. The instability of ELM reduce the efficiency of the extraction and the recovery of the products as well as affecting the recycling liquid membrane. The major concern of this study is to increase the stability of emulsion liquid membrane using green-based liquid membrane as it has been reported to be a promising method for phenol removal (Othman *et al.*, 2017). In order to enhance the membrane stability, the blended surfactant ionic liquid is chosen in this study due to its promising results in increasing the stability of emulsion membrane in the past studies (Lu and Rhodes, 2000; Balasubramaniam and Venkatesan, 2012). According to the method, the surfactant and co-surfactant or ionic liquid or both is mixed during water in oil (W/O) emulsion preparation. It is expected that using blended surfactant in green-based liquid membrane could reduce the membrane swelling and breakage as well as increasing the emulsion lifetime. In addition, by investigating parameters which affect the primary emulsion stability could enhance the ELM process.

Furthermore, most ELM processes for organic compound removal used petroleum based diluent such as kerosene (Jiao *et al.*, 2013), n-hexane (Chanukya and Rastogi, 2013a), 1-octanol (Lee, 2011) and dichloroethane (Chakrabarty *et al.*, 2010). Despite of the expensive materials, the use of petroleum based materials could have significant impact to the environment and is not sustainable for the global green technology. In order to promote green emulsion liquid membrane which is more environmental friendly, the petroleum based diluent can be replaced with renewable sources such as vegetable oils. Vegetable oils are biomass that is non-toxic and biodegradable and according to Venkateswaran and Palanivelu (2006), palm oil is the best diluent for green liquid membrane. In addition, palm oil was found to work well for extraction of phenol using supported liquid membranes (Venkateswaran and Palanivelu, 2006) and bulk liquid membrane (Chang *et al.*, 2011). Therefore, palm oil was selected in this study as the alternative and renewable organic diluent in liquid membrane formulation for recovery of phenol using ELM process.

1.3 Research Objectives

The objectives of this research are:

i. To formulate green emulsion liquid membrane for phenol extraction using blended surfactant ionic liquid.

ii. To determine the parameters affecting primary emulsion stability in ELM process such as HLB value blended surfactant concentration, homogenizer speed, emulsification time, and organic to internal ratio.

iii. To evaluate and optimize the performance of phenol extraction in ELM process using RSM.

1.4 Research Scopes

In order to achieve the first objective, liquid membrane was formulated in terms of liquid membrane components selection. Liquid-liquid extraction process was carried out to determine the suitable types of diluent and stripping agents to be used for phenol removal during the screening process. Several parameters were investigated such as ratios of palm oil to kerosene, carrier type and stripping agent type as well as parameters such as time and rotation speed which were fixed. The amount of phenol extracted was recorded. After finding the optimum ratio of diluent for phenol extraction, screening process for both carrier type and stripping agent type was carried out.

The emulsion liquid membrane system was developed using the selected diluent as well as the carrier and stripping agent. During the water in oil (W/O) emulsion preparation, the blended surfactant was used and tested by dissolving it in the organic diluent in order to develop the liquid membrane. The surfactants that were used are Span 80, Tween 80 as co-surfactant and 1-Butyl-3-metylimidazolium as ionic liquid. The stability of primary emulsion was investigated in the second objective by manipulating the portion of different types of surfactant of Span 80 and Tween 80. Then, parameters incorporated with the emulsion stability were studied such as hydrophile-lipophile balance (HLB) range (4.3-9), blended surfactant concentration (1 -7% w/v), homogenizer speed (5000 – 10000 rpm), emulsifying time (1 – 10 min) and organic to internal (O/I) ratio (1:1 – 3:1). Stability of the emulsion in term of emulsion breakage was recorded.

After selecting the optimum for stable W/O emulsion, several parameters that affect the extraction efficiency of phenol such as agitation speed (150 – 350 rpm), ionic liquid concentration (0.01 – 0.2% w/v), extraction time (1 – 7 min) and treat ratio (1:3 – 1:10) were investigated to identify an optimum extraction condition using the response surface methodology (RSM). Lastly, the optimum condition obtained was tested for validation to fulfill the third objective.

1.5 Significance of Study

Phenol increment in wastewater from various industrial activities has making it crucial for the industry to extract and remove the phenol before discharging wastewater. The removal of phenol from wastewater has become a great concern. Emulsion liquid membrane (ELM) provides a promising alternative separation technology for wastewater treatment with a major limitation, which is emulsion stability. In this research, blended surfactant was used to formulate the emulsion liquid membrane and significantly increase the emulsion stability. Besides, the use of palm oil as the main diluent will promote a more environmental friendly process. Thus, this study aims to increase the stability of emulsion liquid membrane for phenol removal, which could significantly contribute in wastewater treatment and environmental conservation.

1.6 Thesis Outline

This thesis consists of five separately presented chapters. In Chapter 1, research background, problem statement, research objective, research scope and significant of study are introduced. The details of researches related to liquid membrane technology including types, characteristic, current and future development of liquid membrane technology are reviewed in Chapter 2. Details on the previous phenol wastewater studies and their conventional alternatives in extracting and recovering are also reviewed in this chapter. Chapter 3 explains the methodology involved to conduct the study while all the results of the finding are discussed in Chapter 4. Lastly, Chapter 5 suggests the conclusion and recommendation for future study.

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