SYNERGISTIC FORMULATION FOR REACTIVE DYES EXTRACTION USING BENIGN BASED EMULSION LIQUID MEMBRANE

HILMI BIN ABDUL RAHMAN

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Faculty of Engineering
Universiti Teknologi Malaysia

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Specially dedicated to my beloved parents, family members, supervisor and friends. Your endless love, support and prayers make it possible.

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ABSTRACT

Dye loaded in the wastewater is considered as a pollutant due to its toxicity and carcinogenic effect. Hence, the removal of dyes from wastewater is highly demanded. There are many methods introduced for the extraction of dyes but they consume high energy and chemicals. Emulsion liquid membrane (ELM) extraction is one of the promising techniques to simultaneously extract and recover solute from wastewater solution. However, it has some drawbacks such as slow extraction rate, low loading capacity and use of non-friendly based diluent. In this research, emulsion liquid membrane was studied as an alternative method to extract and recover the dye from wastewater. The aims of this study are to formulate a synergistic extractant liquid membrane on extraction of dye (Orange 3R) using palm oil as a diluent, to study the mechanism of Orange 3R extraction and stripping process and optimization of ELM extraction of Orange 3R. The liquid membrane formulation was investigated on the selection of the extractant and synergist extractant using liquid-liquid extraction process. The mechanism of extraction and stripping process was determined by selection of extractant and synergist extractant concentration, extraction kinetic of dyes and selection of stripping agents and its concentration using liquid-liquid extraction process. ELM process was optimized using response surface methodology for four parameters which were surfactant concentration, treat ratio, agitation speed, and extraction time. The important parameter affecting the recovery process of dye which is initial feed concentration was investigated. Results showed that the liquid membrane formulation was determined using Tricaprylmethylammonium Chloride (Aliquat 336) as an extractant and Di-(2-ethylhexyl)phosphoric acid (D2EHPA) as a synergist extractant. The extraction and stripping process was performed at 0.08 M D2EHPA, 0.1 M Aliquat 336, and 0.1 M NaOH, where 86 and 100% of dyes has been extracted and stripped respectively in liquid-liquid extraction process. The optimum condition for ELM extraction performance was achieved at 3.2 % (w/v) of surfactant concentration (Span 80), 12000 rpm of homogenizer speed, 12 minutes of extraction time, 1:9.8 of treat ratio, and 413 rpm of agitation speed. At this condition, 90 % of dye was extracted and 28% was stripped. Almost 10 times of wastewater volume can be treated by a single volume of emulsion liquid membrane. Therefore, ELM process is a promising technology to separate dye from wastewater while solving environmental problems simultaneously.

ABSTRAK

Pencelup yang terdapat di dalam air sisa dianggap sebagai bahan pencemar disebabkan ketoksikan dan kesan karsinogeniknya. Oleh itu, penyingkiran pencelup dari air sisa adalah sangat diperlukan. Terdapat banyak kaedah yang diperkenalkan untuk pengekstrakan pencelup tetapi ia menggunakan tenaga yang tinggi dan bahan kimia yang banyak. Pengekstrakan emulsi membran cecair (ELM) adalah salah satu teknik yang berkebolehan untuk mengekstrak dan memperoleh bahan larut dari air sisa secara serentak. Walaubagaimanapun, ia mempunyai beberapa kelemahan seperti kadar pengekstrakan yang perlahan, muatan beban yang rendah dan menggunakan pelarut yang tidak mesra alam sekitar. Dalam kajian ini ELM telah dikaji sebagai kaedah alternatif untuk mengekstrak dan memperoleh pencelup dari air sisa. Tujuan kajian ini adalah untuk memformulasi membran cecair ekstraktan bersinergi untuk mengekstrak pencelup (Orange 3R) menggunakan minyak kelapa sawit sebagai bahan pelarut, mengkaji mekanisma pengekstrakan Orange 3R dan proses pelucutan, dan pengoptimuman ELM bagi pengekstrakan Orange 3R. Formulasi membran cecair diselidik ke atas pemilihan ekstraktan dan ekstraktan bersinergi menggunakan proses pengekstrakan cecair-cecair. Mekanisma pengekstrakan dan proses pelucutan ditentukan oleh pemilihan kepekatan ekstraktan dan ekstraktan bersinergi, kinetik pengekstrakan pencelup dan pemilihan agen pelucutan dan kepekatannya menggunakan proses pengekstrakan cecair-cecair. ELM dioptimumkan menggunakan kaedah tindakbalas permukaan bagi empat pembolehubah iaitu kepekatan surfaktan, nisbah rawatan, kelajuan pengaduk, dan masa pengekstrakan. Pembolehubah penting yang mempengaruhi proses perolehan pencelup iaitu kepekatan air sisa awal telah disiasat. Keputusan menunjukkan bahawa formulasi membran cecair adalah dengan menggunakan tricaprilmetilammonium klorida (Aliquat 336) sebagai ekstraktan dan asid di-(2-etilheksil)fosforik asid (D2EHPA) sebagai ekstraktan bersinergi . Mekanisma dan proses pelucutan dicapai pada 0.08 M D2EHPA, 0.1 M Aliquat 336, dan 0.1 M NaOH, di mana 86 dan 100% pencelup masing-masing telah diekstrak dan dilucutkan dalam proses pengekstrakan cecair-cecair. Keadaan optimum untuk prestasi pengekstrakan ELM dicapai pada 3.2% (w/v) kepekatan surfactant (Span 80), 12000 rpm kelajuan penghomogen, 12 minit masa pengekstrakan, nisbah rawatan 1:9.8, dan 413 rpm kelajuan pengaduk. Pada keadaan ini, 90% pencelup telah diekstrak dan 28% telah dilucutkan. Hampir 10 kali jumlah air sisa boleh dirawat dengan satu isipadu emulsi membran cecair. Oleh itu, proses ELM merupakan teknologi yang berkebolehan untuk memisahkan pencelup dari air sisa buangan disamping pada masa yang sama dapat menyelesaikan masalah alam sekitar.

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LIST OF ABBREVIATIONS

Aliquat 336	-	Tricaprylmethylammonium Chloride
ANOVA	-	Analysis of Variance
BLM	-	Bulk Liquid Membrane
BBD	-	Box-Behnken Design
D	-	Distribution
D2EHPA	-	Di-(2-ethylhexyl)phosphoric Acid
DF	-	Degree of Freedom
DOE	-	Design of Experiment
ELM	-	Emulsion Liquid Membrane
F	-	Fisher
HLB	-	Hydrophile-lipophile Balance
LLE	-	Liquid-liquid Extraction
MS	-	Mean Square
NaHCO	-	Sodium Bicarbonate
NaOH	-	Sodium Hydroxide
NaCl	-	Sodium Chloride
Na_2CO_3	-	Sodium Carbonate
O/W	-	Oil in Water
RSM	-	Response Surface Methodology
SS	-	Sum-Square
SC	-	Synergistic Coefficient
Span 80	-	Sorbitan Monooleate
SLM	-	Supported Liquid Membrane
TDA	-	Tridodecylamine

TOA	-	Trioctlyamine
TOPO	-	Tri-n-octylphosphine Oxide
TBP	-	Tributlphosphate
TDA	-	Tridodecylamine
UV	-	Ultra-Violet
W/O/W	-	Water in Oil in Water
W/O	-	Water in Oil

LIST OF SYMBOLS

M	-	Molar
rpm	-	Rotation per minutes
w/v	-	Weight per volume
Ppm	-	Part per million
g/mL	-	Gram per milliliter
mg/l	-	Milligram per liter
nm	-	Nanometer
mL	-	Milliliter
min	-	Minutes
hr	-	Hour
TR	-	Treat ratio
°C	-	Degree celcius
cm^{-1}	-	Per centimeter
[]	-	Concentration
µ	-	Viscosity
%	-	Percentage
C_t	-	Concentration of dye at time t
C_{int}	-	Concentration of dye at initial state respectively
k_{eq}	-	Equilibrium constant

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Currently, the world faces critical environmental issues. The main reasons of these issues are rapid growing population, fast growth of industrial development and urbanization. There are various sources that contaminate the water such as domestic and industrial effluents. Industrial waste effluent is a major source for this problem and need to be discharged properly. One of the major sources of wastewater is from the textile industry.

Wastewater containing dyes from the textile industry is considered a pollutant to the environment due to the carcinogenic effect and toxicity. These effluents will cause harmful effect on water quality that will increase in colour, intensity, toxicity and turbidity. It has been predicted that more than 100,000 types of commercial dyes including reactive dyes are produced with over 7 x 10^5 tons produced yearly (Bahloul *et al.,* 2016). Reactive dyes are soluble in aqueous solution and form complexes that possess a remarkable electro-catalytic property. Normally, 4% of the reactive dyes in the textile industry are lost (Bahloul *et al.,* 2013). This is the main concern about the abnormal colouration that has been discharged to the environment which will cause a reduction of photosynthesis on algal and aquatic plants due to limited transmittance of light into water bodies.

There are many methods of dye treatment in wastewater that include physical treatment, chemical treatment and microbiological methods (Forgacs *et al.,* 2004). Although physical technology can be used for the extraction of reactive dyes from aqueous solution like adsorption on activated charcoal, precipitation by alum, ozonation, and electrolysis, it has a drawback of generating a secondary pollution by creating a large amount of sludge (Kabra *et al.,* 2011), quite expensive and have limited their application (Ahmad and Rahman, 2011). Many biological technologies especially on bioremediation methods have been applied on extraction of dyes from textile effluent. Bioremediation is the used of living organism for cleaning the contaminated sites. Previous study shows that many microorganisms are capable of degrading textiles dyes including fungi (Parshetti *et al.,* 2006), yeast (Jadhav and Govindwar, 2006), algae (Daneshvar *et al.,* 2007) and bacteria (Tamboli *et al.,* 2010). The disadvantages of these methods are only focused on the extraction of dye but not to recovered them. Therefore, they produce a secondary pollution.

At the present time, liquid membrane technology is one of the promising methods for separation of specific solute in variety of mixtures. There are three types of liquid membrane which are supported liquid membrane (SLM), bulk liquid membrane (BLM) and emulsion liquid membrane (ELM). Among the techniques, ELM offers some benefits such as low cost, low energy used due to its recycling usage and a high interfacial area resulting in enhancement of mass transfer rate (Peng *et al.,* 2012). The advantages of ELM are the extraction and recovery occur in a one stage process that make product can be separated and concentrated simultaneously. This also has been proved by several researchers on high efficiency of extraction and recovery of various solute from wastewater such as chromium (Othman *et al.,* 2016), tungsten (Lende and Kulkarni, 2015), cadmium (Zeng *et al.,* 2016), acetic acid (Lee, 2015), dyes (Liu *et al.,* 2016), phenol (Othman *et al.,* 2016), palladium (Noah *et al.,* 2015) and rare earth (Zhang *et al.,* 2016). The uniqueness of this system is that it is a simultaneous extraction and recovery in single step process, that separate and concentrate the product in a one step process. However, the main disadvantage of this method is the emulsion instability for longer extraction time. In order to solve the problem and for a faster extraction, a new formulation of liquid membrane need to be explored to overcome the problem.

1.2 Problem Statement

Dye in the textile effluents is a major pollutant due to the toxicity and carcinogenic effect. Even though dyes are considered as a pollutant, dyes are also a valuable chemical in the textile industry and it is a crucial to treat the dye waste water. There are many methods on the extraction of dye in textile industry such as biological (Daneshvar *et al.,* 2007), liquid-liquid extraction, steam distillation, adsorption and chemical oxidation (Villegas *et al.,* 2016). These conventional methods have its own drawbacks such as high operating cost, produces secondary pollutant and time consuming. At the present time, liquid membrane technology especially ELM technique is expected to be the promising method to encounter the disadvantages of conventional methods.

ELM is a combination of two systems which are extraction and stripping process occurred simultaneously in a single stage process. The extractant will extract the solute from the feed phase and form a complex in the liquid membrane phase and diffuse to the interphase of stripping and liquid membrane phase. ELM provides many advantages such as low concentration of chemical used, high surface area for mass transfer, very selective to the targeted solute and low-cost process. Therefore, the ELM was chosen as the great technology to replace the conventional methods on extraction and recovery of dyes in this study. Several researchers attempted on the extraction of dye using ELM process such as the extraction of Methylene Blue (Bahloul *et al.,* 2013b), Congo Red (Dâas and Hamdaoui, 2010), Acid Yellow 99 (Bahloul *et al.,* 2016), Cristal Violet (Agarwal *et al.,* 2010) and Red3BS (Othman *et al.,* 2011). Despite of the advantages, ELM has some problems with the extraction rate and stability that make it unable to be commercialized for industrial purposes.

The extraction rate is associated with the liquid membrane (LM) formulation which is the most important step in ELM process. Suitable extractant, diluent and stripping agent should be chosen to formulate LM for extraction and recovery of dyes. Many previous studies used various petroleum based diluents such as heptane (Chanukya and Rastogi, 2013; Dâas and Hamdaoui, 2010), pentanol (Mokhtari and

Pourabdollah, 2013), hexane (Bahloul *et al.,* 2013b) and kerosene (Masu *et al.,* 2005; Othman *et al.,* 2011; Peng *et al.,* 2012) in LM formulation. Petroleum based LM is non-environmental friendly and can cause other pollutions. Therefore, green based LM such as palm oil and coconut oil has been prove as a potential diluent in many research such as in the extraction of chromium (Othman *et al.,* 2016), Direct Yellow 27 (Melo *et al.,* 2015) and Astacyl Golden Yellow (Muthuraman and Palanivelu, 2006).

On the other hand, the extractant plays the most important role in LM formulation. Various single extractants have been used for extraction of anionic, neutral and cationic species of dyes. However, single extractant has suffered from some drawbacks such as slow phase separations and tiny loading capacity (Zhang *et al.,* 2016a). Therefore, a new formulation was developed by mixing the extractants (synergistic extractant) and this system has been applied on numerous extraction of metal and it has been proven that by using the synergistic extractant the extraction efficiency was increased and reduced the extraction time (Wannachod *et al.,* 2016). According to the finding, a synergistic extractant is expected to be used as an alternative formulation in ELM process for extraction of basic dyes from wastewater.

1.3 Objectives

The main objective of this research is to study the possibility of using a synergistic extractant in ELM system to extract dyes from the wastewater. In summary, the objectives of this research are:

i. To determine a synergistic liquid membrane formulation for Orange 3R dye extraction using Liquid-liquid Extraction (LLE) process.
ii. To investigate the mechanism of Orange 3R extraction and stripping process.
iii. To optimize the extraction of Orange 3R dye in Emulsion Liquid Membrane (ELM) process using RSM.

1.4 Scopes of the Study

In order to achieve the first objective, a liquid membrane formulation has been obtained by using synergistic extractant for the extraction of Orange 3R from the simulated wastewater. The extraction of dyes has been carried out by using a liquid-liquid extraction process. Palm oil has been set as a diluent. Various types of extractant and synergist extractants has been investigated on the performance of extracting dyes. The parameters that has been studied were selection of extractant and synergist extractant.

The mechanism of the Orange 3R extraction process has been carried out by manipulating numerous parameters such as effect of extractant concentration, effect of synergistic concentration and extraction kinetic of dye. Meanwhile, for the stripping process, the parameters studied was stripping agent selection and stripping agent concentration.

The performance of Orange 3R extraction in ELM process has been studied by manipulating several parameters which are treat ratio (1:1-1:10), agitation speed (150-450 rpm), Span 80 concentration (1-7 %w/v) and extraction time (1-15 min) in the second objectives. The optimization of the extraction of Orange 3R dye performance has been designed using design of experiment (DOE) and optimized using RSM method. Four parameters have been chosen such as treat ratio, agitation speed, Span 80 concentration and extraction time to the respond of dye extraction performance.

1.5 Significance of Study

According to the environmental and economic problems, it is important to remove and recover reactive dyes from textile industry wastewater. Liquid membrane technology especially ELM process shows a promising method for separation of organic and metal extraction. ELM process is a promising method that can give a high efficiency due to the advantages of the process such as simple operations, low operational cost, less chemicals used, large mass transfer area was and the extraction and stripping process occurred simultaneously. So, it is a great technology to overcome the disadvantages of the existing conventional treatment. However, ELM process draws some disadvantages on extraction rate and instability of emulsion. In this research, a synergistic extractant is used to formulate the liquid membrane phase to increase the extraction rate. Meanwhile, Span 80 as a surfactant are used in the ELM process was optimized to overcome the swelling and breakage problem.

1.6 Thesis Outline

This thesis consists of 5 chapters. In Chapter One, the research background, problem statement, research objective, research scope and significance of study are introduced. The details of researches related to liquid membrane technology including the types, characteristics, current and future development of liquid membrane technology are reviewed in Chapter Two. Also, the details of researches related to the dyes wastewater and their conventional alternatives in extracting and recovering are reviewed in this chapter. Chapter Three explains the materials used and methodology involved to conduct the study while all the results of the finding will be discussed in Chapter Four. Conclusion and recommendation are discussed in Chapter Five.

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