EMULSION LIQUID MEMBRANE EXTRACTION OF PALLADIUM FROM SIMULATED ELECTROPLATING WASTEWATER

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To my beloved parents, and especially my husband and son

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ABSTRACT

Recently, electroplating wastewater has become a major concern in terms of environmental problem due to toxicity of hazardous metals. However, the monetary value of precious metals, such as gold and palladium, has become a great concern too nowadays. Due to its special electric conductivity and very limited availability, several methods have been tested to identify the potential methods with high selectivity on precious metal recovery from electroplating wastewater. This study was carried out to recover the targeted metal ion, which is palladium, using emulsion liquid membrane (ELM) process. The research involved four major parts, which were liquid membrane component selection, stability study, extraction and recovery, and palladium extraction in matrices solution. Meanwhile, the ELM system comprised of three liquid phases, which were feed phase, liquid membrane organic phase, and receiving phase. The phases of liquid membrane and receiving were emulsified and dispersed into the feed phase to be treated. The important parameters affecting the membrane stability and the recovery of palladium including emulsifying and extraction time, homogenizer and agitation speed, concentrations of surfactant, carrier and stripping agents, pH of feed phase, and treat ratio were investigated. All experiments were carried out using bath extraction process and the recovery part employed a high voltage demulsifier. The results show that the most stable emulsion with 8% of swelling was achieved at 2% (w/v) of span 80, 3 minutes of emulsifying time, 12000 rpm of homogenizer speed, and 200 rpm of agitation speed. The optimum conditions obtained for the extraction and the recovery processes were at 0.2 M of Cyanex 302, 1.0M thiourea in 1.0M H_2SO_4 of stripping agent, 1:3 treat ratio, pH 3 of feed phase, and 5 minutes of extraction time. At these optimum conditions, the maximum extraction and recovery of the palladium was 97% and 40%, respectively. Therefore, the ELM process has shown great potential in extracting palladium from aqueous solution and industrial application.

ABSTRAK

Kini, sisa penyaduran cecair telah menjadi satu kebimbangan dari segi masalah alam sekitar disebabkan oleh ketoksikan logam-logam berbahaya. Walaubagaimanapun, nilai kewangan logam-logam berharga dalam air sisa penyaduran seperti emas dan paladium juga telah menjadi perhatian pada masa kini. Disebabkan oleh sifat kekonduksian elektriknya yang istimewa dan ketersediaannya yang sangat terhad, beberapa kajian telah dijalankan untuk mencari kaedah-kaedah yang berpotensi yang mempunyai kadar pemilihan yang tinggi terhadap pengekstrakan logam berharga daripada air sisa penyaduran. Kajian ini dijalankan untuk mendapatkan logam sasaran, iaitu paladium, dengan menggunakan proses emulsi membran cecair (ELM). Kajian ini melibatkan empat bahagian utama, iaitu pemilihan komponen membran cecair, kajian kestabilan, pengekstrakan dan perolehan semula, dan pengekstrakan paladium dalam cecair matriks. Sementara itu, ELM terdiri daripada tiga fasa cecair, iaitu fasa suapan, fasa organik membran cecair, dan fasa menerima. Membran cecair dan fasa menerima yang diemulsi dan diserak ke dalam fasa suapan akan dirawat. Beberapa parameter penting yang mempengaruhi kestabilan membran dan perolehan semula paladium seperti masa mengemulsi dan pengekstrakan, kelajuan homogenasi dan pengadukan, kepekatan surfaktan, pembawa dan ejen pelucut, pH fasa suapan, dan nisbah rawatan telah disiasat. Semua kajian telah dijalankan menggunakan sistem pengekstrakan kelompok dan bahagian perolehan semula dilakukan dengan menggunakan penyahemulsi voltan tinggi. Keputusan menunjukkan bahawa emulsi yang paling stabil dengan 8% bengkakan telah dicapai pada span 80 2% (w/v), masa mengemulsi 3 minit, kelajuan homogenasi 12000 rpm, dan kelajuan pengaduk 200 rpm. Keadaan optimum yang diperoleh untuk proses pengekstrakkan dan perolehan semula adalah pada 0.2 M Cyanex 302 sebagai agen pembawa, 1.0 M thiourea di dalam 1.0M H_2SO_4 sebagai agen perlucutan, nisbah rawatan 1:3, pH 3 bagi fasa suapan, dan 5 minit bagi masa pengekstrakan. Pada keadaan optimum ini, pengekstrakan dan perolehan semula yang optimum masing-masing adalah 97% dan 40%. Oleh itu, kaedah ELM adalah berpotensi untuk mengekstrak paladium daripada larutan akues dan berpotensi untuk digunakan di dalam bidang industri.

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LIST OF SYMBOLS

kg	–	kilogram
µm	–	micrometer
m^3	–	metre cubic
M	–	molar concentration
mL	–	millitre
ppm	–	part per million
V	–	Volume (m^3)
rpm	–	rotation per minute
t	–	time (s)
w/w	–	weight per weight
w/v	–	weight per volume
wt	–	weight
%	–	percentage
g/cm^3	–	gram per centimeters cubic
°C	–	degree celcius
G	–	gram
g/mol	–	gram per mol
T	–	Temperature
TR	–	Volume emulsion to volume external phase ratio

LIST OF ABBREVIATION

AAS	–	Atomic Absorption Spectrometer
AC	–	Alternate Current
Ag	–	Silver
Au	–	Gold
BLM	–	Bulk Liquid Membranes
CLM	–	Contained Liquid Membranes
D2EHPA	–	Bis(2-Ethylhexyl)Phosphate
DC	–	Direct Current
DOE	–	Department Of Environment
ELM	–	Emulsion Liquid Membrane
EPA	–	Enviroment Protection Agency
H_2S	–	Hydrogen Sulphide
H_2SO_4	–	Sulphuric Acid
HLB	–	Hydrophilic-Lipophilic Balance
IC	–	Integrated Circuits
ILM	–	Immobilized Liquid Membranes
LM	–	Liquid Membrane
MSP-8	–	Di-2-Ethylhexyl Monothiophosphoric Acid
O/W	–	Oil In Water Emulsion
PCB	–	Printed Circuit Board
Pd	–	Palladium
PGM	–	Platinum Group Metal
Pt	–	Platinum
SLM	–	Supported Liquid Membranes
Span 80	–	Sorbitan Monoleate

TIPAC	–	Technology Information, Forecasting And Assessment Council
USEPA	–	United State Environment Protection Agency
W/O	–	Water In Oil Emulsion
W/O/W	–	Water-In-Oil-In-Water

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Palladium and platinum are normally used to plate semiconductor components in order to improve the component's electrical contact. However, as the metals are rare and very low concentrations in wastewater, the development of effective extraction and recovery processes is seriously required for palladium and platinum. Therefore, an electroplating process wastewater is one of the potential palladium recovery sources.

In Malaysia, rapid growth of the semiconductor industry has accelerated waste generation from electroplating processes, causing serious environmental problems due to the high metal content of wastewater. Although the hazardous waste contributes to only a small percentage of all waste generated by industrialization, their impact can be terrible as they do not only affect the physical environment but also biotic components. From 1995 to 1999, averages of 431,000 tonnes of scheduled waste were produced per year, mostly from electronics, clinical waste from hospitals textile, agricultural and domestic activities, chemical and chemical-related industries, and metal finishing (Consumer's Association of Penang, 2001). The Kualiti Alam plant collected 216,500 tonnes of hazardous and toxic waste for disposal and treatment from 1002 companies by the end of 2000 (Consumer's Association of Penang, 2001).

To minimize the quantity of waste, most of the local semiconductor industries have been converting their electroplating process' waste stream into sludge form. However, some of the sludge generated do not pass the leaching test, hence are not allowed by the government to be sent to landfills. Although local policies encourage the treatment of sludge, there are only a few wastewater treatment services in state. In addition, a growing proportion of the industry's clients require environmental management systems. The industry for these reasons is receiving stress on both sides, from the government and from the industry. Hence, the sludge stored and accumulated at their factory sites give problems in providing space. Additionally, compositional analyses have shown that significant amounts of highly valuable or precious metals such as gold, silver and palladium could be recovered from the liquid waste streams. Therefore, extraction from the stream and reuse of such metals will also yield a significant recovery of income by resale. Thus, "recovery" seems to be a solution to a large part of the current electroplating wastewater problems. The recovery of palladium from electroplating wastewater is attractive due to its various applications in industry together with its high market value. Limited availability of palladium also makes it one of the factors for recovery.

There are many techniques which have been commercially recognized to extract palladium from wastewater. A feasibility study was carried out on Indian almond leaf biomass (*Terminalia catappa L.*) by biosorption to remove palladium and platinum ions from water solution (Ramakul *et al.,* 2012). Swain *et al.* (2010) has done extraction of palladium and platinum from chloride solution by liquid-liquid extraction using Alamine 300 as a carrier and this process has achieved a 99.9% recovery of Pt, although in very dilute solutions. The liquid-liquid extraction method also has been used commercially in separating palladium using di-nooctyl sulphide as a carrier (Rydberg *et al*., 2004). Lee and Chung (2000) investigated ion exchange characteristics for the selective separation of palladium and ruthenium.

However, industries are searching for competing alternative technologies which may conquer some of the weaknesses of these methods. The development of a cheap and simple technology to treat the sludge while also recovering the palladium is required to help the industries solve their environmental problems. Many researchers have established that the emulsion liquid membrane extraction process has great potential and has been reported as an advanced method for separating and concentrating metals. Therefore, the emulsion liquid membrane process has a good opportunity to be the key separation and purification operation in the future of electroplating waste treatment as these processes ensure low energy requirements and high product value. To the best of our knowledge, its application has not yet been studied in depth by any researcher, especially on the emulsion liquid membrane stability.

1.2 Problem Statement

Nowadays, the presence of hazardous waste including inorganic solids or sludge containing metals from industries has become a serious problem all around the world. Metals used for plating are usually costly metals with superior qualities such as palladium, platinum, nickel, gold, silver, and chromium. Focusing on the electroplating industry, if the waste were directly discharged into the natural water system, it will undoubtedly cause a lot of environmental problems. It has been noted that out of the total amount of precious metals used in electroplating, 4% becomes waste in sludge which was spent as washing and electroplating solutions (TIPAC, 2009). Other than that, recovery of palladium from electroplating waste is attractive due to its high market value together with various applications in the industry. Because of the special electric conductivity and limited accessibility properties of palladium, recovering this metal from the electroplating waste solutions is economically interesting.

Currently, Malaysia lacks the treatment technologies especially used for treating the electroplating wastewater and there is very limited information available. According to legislations in Malaysia, electroplating companies can store waste on their premises in proper containers and warehouses. However, delays in construction resulted in limited storage facilities and thus, stockpiling of waste. In order to solve this problem, some of the companies had sent their waste overseas for treatment such as to Japan, Australia and the United States (Malaysia Environmental Quality

(Scheduled Wastes) Regulations, 2007). This might consume high costs in transportation and service charges.

In order to separate the palladium from aqueous solutions, various studies have been recently focused on conventional methods. For example, ion exchange is one of the simple ways to separate palladium (Hubicki and Wolowicz, 2009). A disadvantage to this form of treatment is this method involves high operating costs for the ion exchange unit due to resin costs. Biosorption is another efficient low cost process of palladium ions recovery from aqueous solutions. Since biosorption frequently employs dead biomass, it can eliminate the problem of toxicity environments and the need of nutrient requirement (Volesky, 1990). However, biosorption exhibit a short life cycle. Solvent extraction has become an effective technique in the recovery and separation of palladium (Rydberg *et al*., 2004; Swain *et al.,* 2010). However, various problems have been associated with solvent extraction systems such as the corresponding hydrodynamics related problems, third phase problems, and compatibility issues with the diluent.

In order to realise the recovery, it is vital that an efficient recovery process is developed for the palladium. As an alternative, emulsion liquid membrane is one of the configurations in liquid membrane technology which was chosen in this present work due to several advantages over other methods, including single stage operation of both extraction and stripping, less energy requirement, ease of functioning, less chemical consumption, low cost factor and large interfacial area. In addition, ELM process can treat palladium even at very low concentrations and the extracted metal will be concentrated more than 10 times of the external phase in recovery phase (Ramazani *et al.,* 2007). ELMs allow a highly selective transport and efficient enrichment of palladium ions through a very thin liquid membrane. ELM has been intensively investigated and demonstrated as an effective alternative technology for separation and purification process for precious metal extraction such as silver (Othman *et al.,* 2006), gold (Kargari *et al.,* 2006), and palladium (Kakoi *et al.,* 1996).

1.3 Research Objectives

The main objective of this research is to study the feasibility of emulsion liquid membrane (ELM) process to recover palladium from simulated electroplating wastewater. The objectives of this research are as the following:

i. To formulate liquid membrane formulation for palladium extraction from simulated electroplating wastewater.
ii. To study mass transfer mechanism of palladium extraction in ELM.
iii. To investigate the membrane stability and the parameters affecting the extraction and recovery of palladium.

1.4 Research Scopes

To formulate, the study focused on the selection of liquid membrane components. Thus, a screening process was carried out using liquid-liquid extraction to determine the suitable types of carriers and stripping agents to be used for the palladium ions. During the experiments, different types of carriers such as Diisooctylthiophosphinic acid (Cyanex 302), Bis(2-ethylhexyl)phosphate (D2EHPA), Tryoctyl-amine (TOA), Tridecyl-amine (TDA), Tri-n-octylphosphineoxide (TOPO), Tributyl Phosphate (TBP) were used and the amount of palladium extracted was recorded, while the other parameters such as carrier concentration, rotation speed and time were fixed. After finding the most suitable carrier for palladium, the carrier concentrations were varied to find the best concentration for the carrier to extract the palladium. This screening process will identify a suitable carrier and its concentration of palladium and determine its selectivity in the extraction process. At the same time, suitable stripping agents such as thiourea acidic, hydrochloric acid, sodium hydroxide, thiourea and sulphuric acid were screened out for the loaded carrier-palladium complex extraction. Kerosene, chloroform, palm oil and toluene were used as diluents and span 80 as surfactant.

After screening the carrier and strip agent, the emulsion liquid membrane system was developed. There are three components of emulsion liquid membrane; external phase (feed phase), internal phase (stripping solution), and membrane phase (consists of diluent carrier and surfactant).

Others parameters that affect the ELM stability, breakage and swelling were identified in the second objective. Investigation on the stability of primary emulsion was carried out by manipulating the concentrations of surfactant (1 to 7 % w/v), emulsifying (1 – 10 minutes), homogenizer speeds (8000 to 13500 rpm) and agitation speeds (200 – 350 rpm).

The parameters such as treats ratio of emulsion to external phase (1:3 – 1:10) that affect the mass transfer area of extraction process; carrier concentration (0.001 – 0.7 M), and membrane viscosity were studied in this research to establish optimum extraction conditions. After obtaining optimum conditions for palladium extraction and recovery, these conditions were tested for the removal of palladium from complex matrices which are electroplating wastewater. This study fulfilled the third objective.

1.5 Significance of Study

Liquid membrane (LM) separation provides a potentially promising method for effecting various separation operations. The main advantage of this process compared to conventional processes is the extraction and recovery of the solute ion happens simultaneously in one single stage operation. In addition, it has some attractive features, which are simple operations, high efficiency, larger interfacial area and the use of less chemicals, reducing operation costs. In this research, ELM was used to treat palladium in wastewater, which has a high value in the market. Palladium is a rare precious metal with unique physical properties used in diverse industrial applications and in jewellery. Due to its economic value and its limited natural resources, palladium recovery from secondary resources has assumed a great significance.

1.6 Thesis Outline

This thesis consists of 5 chapters, which are presented in this research in sequential order. The research background, problem statement, research objective and research scope are introduced in chapter one. Chapter two reviews the details of researches related to the palladium process in electroplating and their alternatives in extracting and recovering. ELM components and the future development of ELM process is also reviewed in chapter two. As for chapter three, the materials used and methodology involved in this study were stated. All the results and discussion about the findings are presented in chapter four. Lastly, the conclusion and recommendation for further study are presented in chapter five.

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