

RECOVERY OF PALLADIUM FROM AQUEOUS SOLUTION USING
SURFACTANT MEDIATED PRECIPITATION PROCESS

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*Dedicated to VVIP of my life
my beloved mother, Siti Arba'yah Bt Miskam,
my beloved late father, Allahyarham Hj. Buhari B. Hj. Padlan
and siblings, Junainah, Nor Azmi, Nor Azuan, Nor Azam*

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ABSTRACT

In recent years, the demand for precious metals has increased in most emerging countries. The precious metals are mainly used not only in jewellery but also in electrical devices, medical instruments and as catalysts. They can be primarily recovered not only from the ores but also secondary sources like electronic wastes and spent catalysts. This research aimed to develop a method for the recovery of palladium (Pd) from synthetic and real Pd solutions using surfactant mediated precipitation process. The surfactant used was cetyltrimethylammonium bromide (CTAB), while the leaching agents used were hydrochloric acid (HCl), nitric acid (HNO₃): sulphuric acid (H₂SO₄), hydrochloric acid (HCl): hydrogen peroxide (H₂O₂) and aqua regia. The real Pd solution was prepared using selected leaching agents and spent catalyst. The effect of process parameters such as leaching agent types, CTAB concentrations, Pd concentrations, temperature, and contact time on Pd recovery efficiency were studied. Comparative study between CTAB and other reducing agent (i.e. formic acid) was also carried out. The concentration of Pd was determined using atomic absorption spectroscopy (AAS), while the CTAB concentration was determined by the two-phase titration method. The experimental results show that the Pd recovery efficiency strongly depends on the CTAB and Pd concentrations. The increase of temperature resulted in lowering the Pd recovery efficiency. The precipitation process was relatively fast (less than 3 minutes). The Fourier transform infrared (FTIR) spectroscopy and X-ray photoelectron spectroscopy (XPS) analyses showed changes in CTAB functional groups after the precipitation process. The stoichiometry reaction between Pd and CTAB as given by the molar ratio of Pd to CTAB was 2. The results from the leaching process of spent catalyst using several leaching agents (i.e. HCl, HNO₃:H₂SO₄, aqua regia) show that HCl as the leaching agent gave high selectivity towards Pd compared to other metals present in the spent catalyst. It was found that 85% Pd could be recovered from the real Pd solution (i.e. spent catalyst leaching solution) using 1 mM CTAB.

ABSTRAK

Sejak kebelakangan ini, permintaan logam berharga telah meningkat di negara-negara yang membangun. Logam berharga kebanyakannya digunakan bukan sahaja sebagai perhiasan tetapi juga dalam peranti elektrik, alatan perubatan malahan sebagai pemangkin. Selain itu, ia bukan sahaja boleh diperolehi daripada bijih tetapi juga sumber sekunder seperti buangan elektronik dan pemangkin terpakai. Oleh itu, kajian ini dijalankan untuk membangunkan satu kaedah bagi perolehan paladium (Pd) daripada larutan menggunakan proses pemendakan melalui pengantara surfaktan. Surfaktan yang digunakan dalam kajian ini ialah setiltrimetil ammonium bromida (CTAB) manakala ejen pengurusan yang digunakan ialah asid hidroklorik (HCl), asid nitrik (HNO_3), asid sulfurik (H_2SO_4), asid hidroklorik (HCl): hidrogen peroksida (H_2O_2) dan akua regia. Larutan Pd sebenar telah disediakan menggunakan ejen pengurusan yang terpilih dan pemangkin terpakai. Kesan daripada parameter proses seperti jenis ejen pengurusan, kepekatan CTAB, kepekatan paladium (Pd), suhu dan masa sentuhan ke atas prestasi perolehan telah dikaji. Kajian perbandingan antara CTAB dan ejen pengurangan yang lain (asid formik) juga telah dilaksanakan. Kepekatan Pd telah ditentukan dengan menggunakan spektroskopi penyerapan atom (AAS) manakala kepekatan CTAB ditentukan menggunakan kaedah pentitratan dua fasa. Keputusan eksperimen menunjukkan kecekapan perolehan Pd sangat bergantung kepada kepekatan CTAB dan Pd. Peningkatan suhu menyebabkan penurunan kecekapan perolehan Pd. Proses pemendakan telah berlaku dengan pantas (kurang dari 3 minit). Analisa daripada spektroskopi inframerah transformasi Fourier (FTIR) dan spektroskopi fotoelektron sinar X (XPS) menunjukkan perubahan pada kumpulan berfungsi CTAB selepas proses pemendakan. Tindak balas stoikiometri antara Pd dan CTAB yang diberikan oleh nisbah molar Pd kepada CTAB adalah 2. Keputusan proses pengurusan pemangkin terpakai menggunakan beberapa ejen pengurusan (cth. HCl, HNO_3 : H_2SO_4 , akua regia) menunjukkan dengan menggunakan HCl sebagai ejen pengurusan, pemilihan ke atas Pd lebih tinggi berbanding dengan logam lain yang terdapat di dalam pemangkin terpakai. Dalam kajian proses perolehan logam melalui larutan pengurusan pemangkin terpakai, 85% Pd boleh diperolehi menggunakan 1 mM CTAB.

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LIST OF ABBREVIATIONS

AAS	-	Atomic Absorption Spectrophotometer
ACF	-	Adsorbing Colloid Flootation
Ag	-	Silver
Au	-	Gold
BWN	-	Bleached waste newspaper
CMC	-	Critical micellar concentration
CPC	-	Cetyl pyridinium chloride
CPE	-	Cloud point extraction
CPT	-	Cloud point temperature
CTAB	-	Cetyltrimethylammonium bromide
ELM	-	Emulsion Liquid Membrane (ELM)
FTIR	-	Fourier Transform Infra-Red
HLLW	-	High Level Liquid Waste
MAC	-	Maximum allowed concentration
MEUF	-	Micellar-enhanced ultrafiltration
OTAB	-	Octadecyl trimethyl ammonium bromide
PCB	-	Printed circuit board
Pd	-	Palladium
PEG	-	Polyethylene glycol
PGMs	-	Platinum group metals
PM	-	Precious metal
Pt	-	Platinum
PWB	-	Printed wiring board
REEs	-	Rare earth elements
SDS	-	Sodium dodecylsulphate
TTAB	-	Tetradecyl trimethyl ammonium bromide

WEEE	-	Waste electric and electronic equipment
WN	-	Waste newspaper
WPCB	-	Waste printed circuit board
XPS	-	X-ray photoelectron spectrophotometer
XRF	-	X-ray fluorescence spectrophotometer

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CHAPTER 1

INTRODUCTION

1.1 Research Background

The peculiar physical and chemical properties of precious metals have attracted numerous industries. Fujiwara *et al.* (2007) stated that platinum group metals (PGMs) are greatly used in various fields. One example is as the catalyst in many chemical process, electrical and electronic devices, medicine and jewellery. Despite the continuous demand of these materials, the consumption of precious metals by the industry is growing and is expected to increase further in years to come. The scarcity of precious metals and their increasing demands have encouraged many studies to recover these precious metals for a sustainable development.

Due to the fact that these metals occurred in small amounts on earth, they must be effectively recovered from various wastes for recycle and reuse purposes (Bernardis *et al.*, 2005). From the economic point of view, the recovery process should be highly selective towards the precious metals as to separate the metal from base metals such as copper, iron, and zinc that often coexist with the precious metals in disproportionate amounts. The conventional and traditional processes used for separating and refining precious metals consist of a series of steps involving dissolution of aquaregia, conditioning, and then precipitation, of which are inefficient in terms of the degree of purification, yield, operational complexity, energy consumption, and labor costs.

Two sources for valuable metal recovery are from the primary sources (ore) and from secondary sources like electronic scraps and industrial wastes. Besides, precious metals can also be recovered from spent catalysts. In recent years, the amount of spent hydroprocessing catalysts produced throughout the world was in the range of 150,000–170,000 tons year⁻¹ (Dufresne, 2007). A stable growth in the processing of heavier feedstock is estimated to increase the generation of spent catalysts in the coming years because of the instant growth in diesel hydrotreating capacity, such that is to meet the increasing demand for cleaner fuels with ultralow sulphur levels. For instance, it is projected that in Kuwait alone, the generation of spent catalysts will be more than double in the near future in which approximately, 75% of the all industrial chemical processes are based on catalysis (Dufresne, 2007). Supported metal catalysts are also widely used in many chemical processing industries. Thus, spent catalysts are a potential source of the contained critical metals.

Unfortunately, spent catalysts fall under the category of hazardous industrial waste and their disposal is a problem (Kumar *et al.*, 2013). The treatment of spent catalysts has gained importance recently owing to two reasons which are, the metal values present and the need for safe disposal to avoid environmental pollution. In addition, precious metals can easily transfer via root plants into biological material. It also gives harmful effects towards human health, of which may cause eye irritation, primary skin burning and restrain enzyme activity. Hence, precious metal recovery has become economically and environmentally very important (Awual *et al.*, 2014).

Being significantly crucial, this issue has triggered numerous scientific inquiries on the development of methods for pre-concentration of precious metals and their selective recovery. Lopes *et al.* (2012) stated that the main technologies that have been developed for precious metal recovery from its solutions include adsorption (Xiong *et al.*, 2009), ion exchange (Liang *et al.*, 2013), cementation, solvent extraction (Young *et al.*, 2010), electrodeposition (Giridhar *et al.*, 2006), membrane processes, precipitation (Pakarinen and Paatero, 2011), electrolytic recovery and reductive exchange. Of those, adsorption, precipitation and ion-

exchange were claimed the most favourable technologies available for metal recovery.

Fujiwara *et al.* (2007) stated adsorption method is effective for pre-concentration and separation of gold from aqueous solutions. Instead, Matsubara *et al.* (2000) claimed, as the solution also comprise large quantities of other metals the adsorption of precious metals from acidic chloride solution is a challenging task in term of its selectivity. Along with that, adsorption or solvent extraction is constrained by elution or stripping for the recovery of precious metals from hydrochloric acid. Xiong *et al.* (2009) found that chemically modified persimmon waste as the adsorbent could improve the selectivity of precious metal recovery. Nonetheless, in order to extract tannin from feed materials, a high cost is needed. In addition, the persimmon waste gel has a slow kinetic of reduction and adsorption.

Precipitation by biphasic system approach that include micellar extraction with ultrafiltration, polyethylene glycol (PEG) biphasic aqueous system or surfactant liquid membrane system were found to be in advantage due to the non-flammable, inexpensive components and nontoxic process involved. Studies on the use of surfactants for precious metal recovery are rarely found. So far, researchers have studied the recovery of Cr (VI) using cationic surfactant (Jing *et al.*, 2011), and recovery of platinum group metal anions by cationic surfactant using micellar-enhanced ultrafiltration (MEUF) (Ghezzi *et al.*, 2008 and Gwicana *et al.*, 2006). In fact, the micellar-enhanced ultrafiltration (MEUF) method is quite simple, saves time and cost-effective.

1.2 Problem Statement

Precious metal is a widely consumed material not only in jewellery, health care and equipment, but also in many chemical process industries such as petroleum reforming, pharmaceutical and petrochemical. The great demand of the precious metals has brought problems with regards to the supply of raw materials, due to the highly valuable and limited source. Meanwhile, the growing amount of spent

catalysts, along with the electrical and electronic industrial wastes that contained precious metals such as palladium is creating negative impacts upon human and the environment. Hence, recovery of precious metals from secondary sources is one of the ways that can be applied in order to manage the two main concerns regarding precious metals.

The awareness of researches about this problem has brought to the development of numerous methods in recovering precious metals from secondary sources. Previous studies have mentioned many hydrometallurgical methods to recover platinum and palladium in spent petroleum catalysts (Kumar *et al.*, 2013). Other methods being studied for precious metal recovery are as claimed by previous researchers that adsorption, precipitation and ion-exchange are among the preferable methods (Lopes *et al.*, 2012).

Generally, conventional ion exchangers are not sufficiently selective to remove certain metals from large accompanying metals. Meanwhile, solvent extraction is less effective in the recovery of very dilute solutions, and electrolytic process is not preferable for low metal concentration due to the low performances of the process. Recent developments in the studies of metal recovery have led to renewed interest in the use of surfactants. Micellar-enhanced ultrafiltration that was proposed by Ghezzi *et al.*(2008) have managed to remove 90% of palladium (II) ions from water with a cationic surfactant. The function of the surfactants was to attract metal ions prior to separation of the metal-surfactant association through ultrafiltration. Besides, an impregnation of surfactants on the surface of adsorbents enhanced the capacity to adsorb heavy metals (Taylor *et al.*, 2011). In addition, Akita *et al.* (1997) has been using polyoxyethylene nonyl phenyl ether (PONPEs) as a non-ionic surfactant in the MEUF of gold(III) from a diluted hydrochloric acid solution. However, the ultrafiltration membrane could be damaged if a highly acidic solution is used. Another method involving precious metal separation using surfactant is the liquid surfactant membrane (LSM) technique (Kakoi *et al.*, 1996). Thiourea was practically used as the stripping agent for palladium recovery. Though, the use of thiourea has been doubt by some because of its carcinogenic property.

Another method involving precipitation using surfactant was introduced by Talens-alesson and Porrás-rodríguez (1999) which was known as adsorptive micellar flocculation. In this method, cations were bound onto micelle surface and forming flocs to be filtered. This method has been implemented for 2, 4-Dichlorophenoxyacetic acid removal and so far has not yet been applied for palladium recovery. Thus, this research has focused on recovering palladium from aqueous solution with surfactant mediated precipitation process. The surfactant, namely cetyltrimethylammonium bromide (CTAB) was used to precipitate palladium chloro-complex from the solution. The palladium (Pd) chloro-complex is an anionic form, whereas the CTAB is a positively charged surfactant. Generally, at the concentration above CMC value, the CTAB aggregated forming micelles that entrapped the Pd chloro-complexes (i.e. PdCl_4^{2-}). The aggregation eventually increased in size which later precipitated as a result of its decreasing solubility. Compared to the complexity of other surfactant separation techniques, this method was considered simple to implement. The recovery of Pd using CTAB is so far understudied and would potentially become a reliable source in the recovery of other precious metals from leaching solutions in the near future.

1.3 Objectives and Scopes of Research

The objectives and scopes of this research are as follows:

- 1) To study the palladium recovery performance from synthetic solution

In this study, the precious metal used was palladium. The recovery of palladium was carried out using synthetic solution at various experimental conditions. The effect of parameters such as CTAB concentrations, pH, leaching agents and palladium concentrations on recovery performance were investigated. The concentration of palladium was determined using Atomic Absorption Spectroscopy (AAS).

The experimental data were analysed in terms of equilibrium and kinetics of the precipitation process. The stoichiometry of the precipitation reaction was determined. In order to understand the mechanism of Pd recovery process, the analytical techniques such as the Fourier Transform Infrared (FTIR) Spectroscopy and X-ray Photoelectron Spectroscopy (XPS) were used.

2) To investigate palladium recovery performance from leaching solutions

In order to evaluate the palladium recovery performance from a few leaching solutions, the spent catalyst from a local industry was used. The spent catalyst was first characterized with XRF to determine its chemical composition. Various leaching solutions were studied and the metal concentration in the leaching solution was determined by Atomic Absorption Spectroscopy (AAS). The palladium recovery performance from leaching solutions was investigated at various experimental conditions.

1.4 Thesis Outline

This thesis was organized into 5 Chapters. Chapter 1 presents the general information regarding the research which includes introduction, problem background, objectives and scopes of the study, thesis outline and chapter summary. Chapter 2 discusses precious metals and their recovery. A special attention was focused on surfactants and its application for the separation process was reviewed. The research methodology was discussed in Chapter 3 that outlined the materials, experimental procedures for palladium recovery process, and the analytical techniques Chapter 4 exhibits the results and discussions, while Chapter 5 summarizes the conclusion and listed some future research recommendations.

1.5 Summary

The fundamental issue of this study was the recovery of palladium from synthetic solution at various experimental conditions followed by the investigation on palladium recovery from the leaching solution prepared from a spent catalyst. The scientific understanding provided from this research could lead towards the application of the same method for the recovery of various precious metals such as Pd, Ag, Pt and Au from other secondary sources like electrical appliances, photographic waste and automobile catalysts.

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