RECOVERY OF PALLADIUM FROM AQUEOUS SOLUTION USING SURFACTANT MEDIATED PRECIPITATION PROCESS

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A thesis submitted in fulfilment of the requirements for the award of the degree of Master of Engineering (Chemical)

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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
ACKNOWLEDGEMENTS

In the name of Allah the Most Gracious, the Most Merciful and the Most Loving. Peace be upon Prophet Muhammad S.A.W, the messenger of Allah. Alhamdulillah to the Almighty Allah for His guidance and helps throughout my journey in this world. In addition, I would like to express my gratitude to my supervisor, Assoc. Prof. Dr. Hanapi Mat for his supervision, encouragement and knowledge throughout the project period. Certainly, without his continued provision and motivation, this thesis would not have been the same as presented here.

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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.
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<tr>
<td>AAS</td>
<td>Atomic Absorption Spectrophotometer</td>
</tr>
<tr>
<td>ACF</td>
<td>Adsorbing Colloid Floatation</td>
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<tr>
<td>Ag</td>
<td>Silver</td>
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<tr>
<td>Au</td>
<td>Gold</td>
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<tr>
<td>BWN</td>
<td>Bleached waste newspaper</td>
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<tr>
<td>CMC</td>
<td>Critical micellar concentration</td>
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<td>CPC</td>
<td>Cetyl pyridinium chloride</td>
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<td>CPE</td>
<td>Cloud point extraction</td>
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<tr>
<td>CPT</td>
<td>Cloud point temperature</td>
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<tr>
<td>CTAB</td>
<td>Cetyltrimethylammonium bromide</td>
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<tr>
<td>ELM</td>
<td>Emulsion Liquid Membrane (ELM)</td>
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<tr>
<td>FTIR</td>
<td>Fourier Transform Infra-Red</td>
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<td>HLLW</td>
<td>High Level Liquid Waste</td>
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<td>MAC</td>
<td>Maximum allowed concentration</td>
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<td>MEUF</td>
<td>Micellar-enhanced ultrafiltration</td>
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<td>OTAB</td>
<td>Octadecyl trimethyl ammonium bromide</td>
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<tr>
<td>PCB</td>
<td>Printed circuit board</td>
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<tr>
<td>Pd</td>
<td>Palladium</td>
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<td>PEG</td>
<td>Polyethylene glycol</td>
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<td>PGMs</td>
<td>Platinum group metals</td>
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<td>PM</td>
<td>Precious metal</td>
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<tr>
<td>Pt</td>
<td>Platinum</td>
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<tr>
<td>PWB</td>
<td>Printed wiring board</td>
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<tr>
<td>REEs</td>
<td>Rare earth elements</td>
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<td>SDS</td>
<td>Sodium dodecylsulphate</td>
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<td>TTAB</td>
<td>Tetradecyl trimethyl ammonium bromide</td>
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<tr>
<td>Abbreviation</td>
<td>Description</td>
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<tr>
<td>WEEE</td>
<td>Waste electric and electronic equipment</td>
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<td>Waste newspaper</td>
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<td>WPCB</td>
<td>Waste printed circuit board</td>
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<td>XPS</td>
<td>X-ray photoelectron spectrophotometer</td>
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<td>XRF</td>
<td>X-ray fluorescence spectrophotometer</td>
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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 Porras-rodriguez (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.
REFERENCES


