OPTIMIZATION STRATEGIES OF ULTRASONIC-ASSISTED DIGESTION OF PRINTED CIRCUIT BOARD PRIOR TO DETERMINATION OF COPPER AND LEAD

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DEDICATION

This dissertation is dedicated to my late father, who taught me that the best kind of knowledge to have is that which is learned for its own sake. It is also dedicated to my mother, who taught me that even the largest task can be accomplished if it is done one step at a time

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ABSTRACT

Over the past few decades, waste management has become a subject of global interest not only for environmental reasons but also for value-added goods that it provides. The use of hydrometallurgy in electronic waste (e-waste) treatment is effective in metal recovery while at the same time curtailing the waste disposal rate into the ecosystem to some degree. Nevertheless, there is growing concern over the use of concentrated acid due to the correlation of secondary pollution with its disposal. The purpose of the present study is to investigate a suitable method to efficiently recover copper (Cu) and lead (Pb) from waste mobile phone printed circuit boards (PCBs). A hydrometallurgy method was developed for effective metal leaching while maintaining low acid concentrations. Preliminary experiments were conducted to establish a suitable solvent with better dissolution strength for the target metals. Effect of four parameters viz. acid concentration, the molar ratio (HCl: HNO_3), time (min) and temperature (°C) were studied using one-variable-at-a-time (OVAT) and multivariate response surface methodology (RSM) optimization. Results obtained showed that 95% Cu and 86% Pb yield were achieved using the RSM method and remarkably, 107% Cu was recovered and a lesser 91% Pb was recorded via the OVAT method. Atomic absorption spectrometer was used in determination of Cu and Pb, relative standard deviation (RSD) for intra-day precision and inter-day were 5.3%, 5.0% and 12.7%, 6.3% respectively. Limit of detection (LOD) and limit of quantitation (LOQ) obtained were 0.082 µg/g and 0.312 µg/g (Pb) whereas 0.02 µg/g and 0.064 µg/g (Cu) were recorded. Overall, the method demonstrates a significant recovery rate, thus, it could be deployed in the dissolution of metals from e-waste. Although OVAT approach gave better recovery rates, RSM seems to be more practical due to the interaction factor.

ABSTRAK

Sejak beberapa dekad yang lalu, pengurusan sisa telah menjadi subjek kepentingan global bukan sahaja disebabkan faktor-faktor alam sekitar, tetapi juga untuk faedah nilai tambah yang disediakannya. Penggunaan hidrometalurgi dalam rawatan sisa elektronik (e-waste) telah terbukti berkesan dalam pemulihan logam manakala pada masa yang sama mengurangkan kadar pelupusan sisa ke dalam ekosistem pada tahap tertentu. Walau bagaimanapun, terdapat kebimbangan yang semakin meningkat terhadap penggunaan asid pekat disebabkan oleh interaksi pencemaran sekunder daripada hasil pelupusannya. Tujuan kajian ini adalah untuk menyiasat kaedah yang sesuai untuk memulihkan Kuprum (Cu) dan Plumbum (Pb) dengan cekap yang terdapat di dalam bahan buanfan papan litar bercetak telefon bimbit (PCB). Kaedah hidrometalurgi telah dibangunkan untuk mendapatkan kembali logam yang berkesan sambil mengekalkan kepekatan asid yang rendah. Eksperimen awal telah dijalankan untuk menyiasat pelarut yang sesuai dengan kekuatan pencairan yang lebih baik untuk logam sasaran. Empat parameter iaitu kepekatan asid, nisbah molar (HCl: HNO_3), masa (min) dan suhu (°C) dikaji menggunakan Pengukur Kadar optimum pemboleh ubah pada satu masa (OVAT) dan pengukur kadar optimum multivariat Pengukur kadar optimum tindak balas permukaan metod (RSM). Keputusan yang diperoleh menunjukkan bahawa 95% Cu dan 86% Pb dapat dicapai dengan ketara menggunakan kaedah RSM, 107% Cu telah pulih dan kurang 91% Pb telah direkod melalui kaedah OVAT. Untuk penentuan Cu dan Pb, sisihan piawai relatif (RSD) untuk ketepatan dalam masa sehari dan hari berikutnya adalah 5.3%, 5.0% dan 12.7%, 6.3%. Had pengenal pastian (LOD) dan had kuantiti (LOQ) yang diperoleh untuk Pb ialah 0.082 μg / g dan 0.312 μg / g manakala 0.02 μg / g dan 0.064 μg / g dicatatkan untuk Cu. Secara keseluruhannya, kaedah ini menunjukkan kadar pemulihan yang signifikan, oleh itu, ia boleh digunakan dalam pemulihan logam daripada rawatan sisa elektronik. RSM lebih praktikal digunakan di dalam kajian in walaupun pendekatan OVAT memberi kadar pemulihan yang lebih baik disebabkan faktor interaksi.

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LIST OF ABBREVIATIONS

AOAC		Association of Official Agricultural Chemists
ANOVA	-	Analysis of Variance
BBD	-	Box-Behnken Design
BFR	-	Brominated Flame Retardant
CCD	-	Central Composite Design
DOE	-	Design Of Experiment
EC	-	Electronic Component
EEE	-	Electrical and Electronic Equipment
EPA	-	Environment Protection Agency
FAAS	-	Flame Atomic Absorption Spectroscopy
ICH	-	International Council for Harmonisation
IT	-	Information Technology
LOD	-	Limit Of Detection
LOQ	-	Limit Of Quantitation
MPPCB	-	Mobile Phone Printed Circuit Board
PM	-	Precious Metal
PCB	-	Printed Circuit Board
WEEE	-	Waste Electrical and Electronic Equipment

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Industrialization came about through discovery and exploitation of minerals for various uses, from aggregates for constructions, iron used in bulding rails and bridges; aluminium used in automobile and airplane to gold, diamond and sapphire for jewelry and ornaments. Thus, mining of these resouces is what keep the economy of several developed nations around the world afloat. Mineral is defined as a naturally occurring substance found on the surface or in the earth crust that is made up of one or more elements chemically bound together in an orderly and accessible manner ((McMahon and Moreira, 2014; Ericsson and Lof, 2017).

Earth is blessed with numerous mineral resources scattered unevenly around the world, however, increasing population and continuous search for new applications for minerals/elements for economic gains are proportional to the consumption of the resources causing fears of depletion of the less abundant minerals in not distant future (Henckens *et al.*, 2019). The threat mobilizes scientists and experts to look for alternative/secondary sources from which the metals can be obtained. This would go a long way in not only meeting the market demands but also slow down the rate of exhaustion of these valuable metals thereby ensuring their sustenance. Recycling has since been marked as one of the most efficient methods. Electronic waste (e-waste) is the fastest growing waste and arguably the richest in metals/elements content. According to Syed, (2012) concentration of precious metals in the printed circuit boards (PCBs), could be 10 times or higher than what is obtainable from ores.

Electronic waste also known as e-waste can be described as electrical and electronic equipment that reaches its end-of-life. These are discarded units that are no longer in use by consumers. Waste electrical and electronic equipment (WEEE) can be from laptops, hard disc drives (HDD), internet routers, switchboards, television, air-condition, washing machine, personal computers (PCs), mobile phones, etc. (Ogunniyi *et al.*, 2009; Priya and Hait, 2018). PCB is the most common component of these devices and considered the most valuable due to its high metal content.

The rapid generation of electrical and electronic equipment waste is attributed to globalization and revolution in the telecommunication sector. Furthermore, new design, higher capacity, and more features units are being released by different companies in an ever competing market every few months to replace the old versions of electronics. Interestingly, the decreased cost is also the main driver for the fast-growing e-waste. Robinson (2009) reported that the major producers of e-waste in the world are the United State of Ameriaca (USA), Europe and Australia, China and Latin America are also expected to become major players in a global stage in the coming years. The unprecedented pace at which WEEE are being generated is alarming. Nearly 50 million tons are produced annually and the number could potentially doubled in the next 25 to 30 years (Pace *et al.*, 2019).

Waste mobile phones are perhaps the major component that contributes to growing e-waste owing to the new products that come into the market every few months and also a drastic decrease in the life-span of mobile phones. It was estimated that by 2020, waste mobile phone generation could be up to 18 times higher than in 2007 (Tripathi *et al.*, 2012). Waste of mobile phone PCBs composes up to 60 different elements including valuable and toxic substances. It is an exceptionally good candidate for precious metals recovery due to high precious metals (PM) content compared to most small size electrical and electronic devices (Holgersson *et al.*, 2017).

Hydrometallurgy has gained acceptance from laboratory to large scale industrial applications. This is so due to (i) simple-to-follow steps not requiring highly skilled personnel (ii) less energy requirement, (iii) moderate waste generation warranting less costly waste disposal, and (iv) excellent metal recovery (Zhang *et al.*, 2012; Mecucci and Scott, 2002). Among the prevailing leaching reagents, use of aqua regia for metals recovery from e-waste has been widely reported in the literature due to its efficiency in dissolution of wide range of metals present in waste PCBs from precious metals such as gold (Au), silver (Ag), palladium (Pd) to base metals such as copper (Cu), aluminium (Al), zinc (Zn) and hazardous metals viz. lead (Pb), arsenic (As), mercury (Hg), cadmium (Cd) etc. (Kim *et al.*, 2018; Shah, *et al.*, 2014). The aim of this research was to recover Cu and Pb through different optimization method using dilute aqua regia and to compare the concentration of Pb found with the Environment Protection Agency (EPA) allowed limit.

1.2 Problem Statement

Over the past few years, the recycling industry has received a boost primarily due to the explosion in technology seeking to make life easier. Consequently, flooding the markets with gadgets ranging from computers, smartphones, electronic toys, etc. which essential component, PCB contains wide range and high content valuable as well as hazardous metals. The phenomenal rate at which e-waste is generated around the globe poses a grave threat to human health and the environment due to hazardous metals it holds. Pyrometallurgy, a recycling method involving the use of high temperatures usually in a furnace to melt electronic waste into a slurry, allowing easy separation of ferrous from the nonferrous fraction with a high purity metals recovery (Cui and Zhang, 2008) was efficient tool used to tap these metals from e-waste. Nevertheless, a highly toxic by-product from the process is of grave concern given that one of the major drives for recycling is to reduce the level of poisonous metals from e-waste being released into the environment.

Chemical leaching however, is widely adopted due to its efficiency and selectivity. Although, proposed as an alternative leachant due to highly toxic and handling issues of cyanide in Au dissolution from waste material. Yet, aqua regia (HCl: HNO_3) is itself considered corrosive (Akcil *et al*., 2015). It is noteworthy that most of the literature reported on the use of concentrated aqua regia as leaching media (Ernst *et al*., 2003; Kim *et al*., 2018). In this study, we focused on the use of dilute

HCl: HNO_3 media with the aid of ultrasonic bath for recovery of Cu and Pb metals from waste mobile PCBs.

1.3 Objectives of the Study

The objectives of the research are:

i. To develop an ultrasonic bath assisted method for Cu and Pb metals recovery from waste mobile printed circuit board.

ii. To optimize Cu and Pb leaching/dissolution using both one-variable-at-a-time (OVAT) and response surface methodology (RSM) design software and compare the results

iii. To validate each method and establish whether the concentration of Pb in the leachate is within the EPA threshold limit.

1.4 Scope of the Study

Ultrasonic water bath with fixed frequency and heating power 180W was used to provide the needed agitation that would enhance metal dissolution from PCBs originating from different broken mobile phone brands. Diluted mineral acids viz. hydrochloric acid (HCl), sulphuric acid (H_2SO_4), nitric acid (HNO_3), and hydrogen peroxide (H_2O_2) were used in the preliminary analysis to select a suitable solvent for the method development. Mineral acid offer better process control and are relatively inexpensive (Jadhav and Hocheng, 2015) and thus, were chosen in the development of the method.

Four reaction parameters namely; Aqua regia concentration, HCl: HNO_3 molar ratio, leaching time and reaction temperature were optimized using OVAT and RSM design software Box-Behnken Design (BBD).

Leachates obtained following sample preparation were subjected to flame atomic absorption spectroscopy (FAAS) analysis for the determination of Cu and Pb.

1.5 Significance of the Study

This study firstly explores a new method that could highlight yet more efficient metals recovery approach from electronic waste in metallurgical route with the potential to benefit e-waste recycling industries by cutting cost and reduce the environmental concerns that arose from the use of concentrated inorganic acids. Secondly, it will guide into how serious the community should take the issue and by extension help in making informed decisions on illegal dumping of end of life electrical and electronics.

1.6 Conceptual Framework

The stages involved in the proposed hydrometallurgy method for leaching/recovery of Cu and Pb from waste mobile phone PCBs could be seen in Figure 1.1

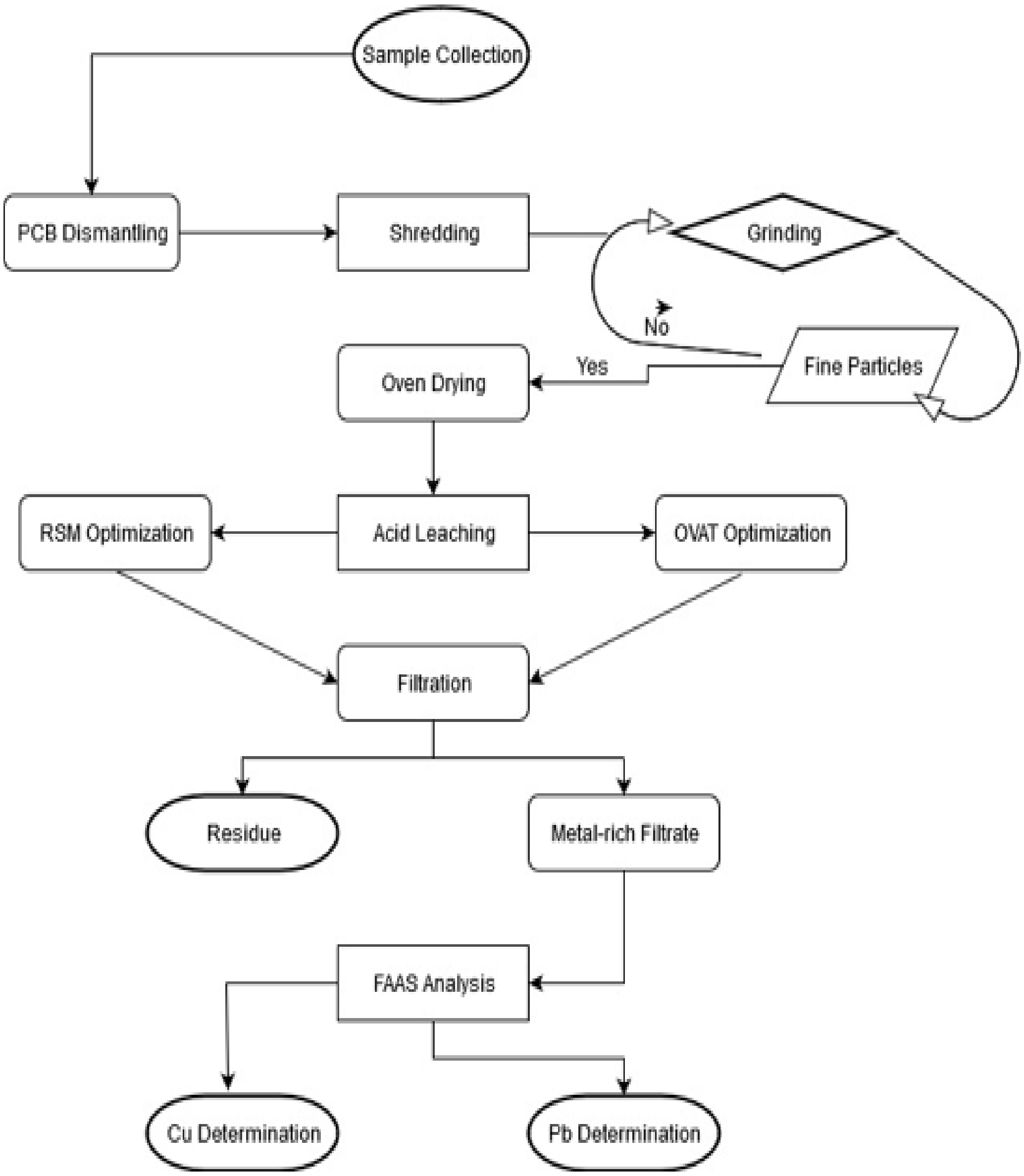

Figure 1.1 Research flow chart

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