

A STUDY ON PHYSICAL SEPARATION PROCESSES
FOR RECOVERY METALS FROM WASTE PRINTED
CIRCUIT BOARDS (PCBs)

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UNIVERSITI MALAYSIA PERLIS
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**A Study on Physical Separation Processes for
Recovery Metals from Waste Printed Circuit Boards
(PCBs)**

By

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A thesis submitted
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DECLARATION OF THESIS

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LIST OF ABBREVIATIONS

AAS	Atomic absorption microscopy
Al	Aluminium
Be	Beryllium
BFR	Brominated flame retardants
Br	Bromine
Cd	Cadmium
Cu	Copper
Fe	Iron
Hg	Mercury
MIBC	Methyl isobutyl carbinol
Ni	Nickel
Pb	Lead
PCBs	Printed circuit boards
PVC	Polyvinyl carbonate
SEM	Scanning electron microscopy
Sn	Tin
W	Tungsten
WEEE	Waste of electric and electronic equipment

LIST OF SYMBOLS

cm	centimetre
°C	degree Celsius
g	gram
g/m ³	gram per cubic meter
kg	kilogram
kg/m ³	kilogram per cubic meter
L	liter
mL	millilitre
mL/s	millilitre per seconds
mm	millimetre
N	Newton
nm	nanometre
RPM	revolutions per minute
s	seconds
Wt%	weight percentage
%	percentage
µm	micrometer

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Kajian tentang Proses Pemisahan Fizikal untuk Perolehan Logam dari Sisa Papan Litar Bercetak (PCBs)

ABSTRAK

Dalam mempertimbangkan peningkatan sisa papan litar bercetak (PCBs), proses pemisahan fizikal telah dijalankan untuk memperolehi logam dari sisa papan litar bercetak. Kajian ini bertujuan untuk mendapatkan perolehan logam dari sisa papan litar bercetak terutamanya kuprum (Cu) secara berkesan dan mesra alam. Proses pemisahan fizikal bermula dengan kominusi untuk menghasilkan partikel saiz yang terkawal. Kemudian, proses pemisahan dibahagikan kepada dua bahagian mengikut kecekapan yang optimum pada julat saiz tertentu. Pecahan saiz $-600+300\mu\text{m}$ dan $-1180+600\mu\text{m}$ dipisahkan melalui pemisahan gravity menggunakan pemisah makmal Mozley. Selepas itu, langkah pengayaan pada pecahan kosentrat dilakukan melalui pemisahan magnetik menggunakan pemisah magnetik gelean nadir bumi. Sementara itu, untuk pecahan saiz $-150\mu\text{m}$, $-300+150\mu\text{m}$ dan $-600+300\mu\text{m}$ dipisahkan melalui pengapungan buih menggunakan sel pengapungan makmal Denver D-12. Pencirian sisa papan litar bercetak dijalankan melalui analisa mikrograf dan analisa unsur. Analisa mutuan mikrograf menggunakan mikroskop stereo-zoom, mikroskop optik dan mikroskop imbasan cahaya. Analisa unsur menggunakan analisis spektrum penyerapan atom (AAS) dan spektrometer serakan tenaga (EDS). Melalui analisa saiz partikel, perolehan maksima pemisahan fizikal dilakukan dengan menyasarkan perolehan dalam empat pecahan saiz julat terkawal; $-150\mu\text{m}$, $-150+300\mu\text{m}$, $-300+600\mu\text{m}$ dan $-600+1180\mu\text{m}$. Penilaian mutuan liberasi pada partikel papan litar bercetak yang halus menunjukkan masih ada partikel yang tidak liberasi pada sisa papan litar bercetak yang halus ($-75\mu\text{m}$). Sebagai unsur logam yang tertinggi di dalam PCBs, perolehan (R) dan nisbah pengayaan (ER) kuprum (Cu) dibincangkan secara mendalam dalam projek ini. Dengan menggunakan pemisah makmal Mozley, perolehan Cu meningkat dari 80.85% (ER 2.07) pada pecahan saiz $-600+300\mu\text{m}$ kepada 89.65% (1.93) pada pecahan saiz $-1180+600\mu\text{m}$ size. Dengan demikian, kecekapan pemisahan gravity meningkat dengan peningkatan saiz partikel. Signifikansi dari perolehan yang makin sedikit pada pecahan saiz yang makin kecil menunjukkan kehilangan logam berharga pada pecahan saiz ini dan menjadi bukti bahawa partikel yang lebih halus ($-300\mu\text{m}$) tidak sangat berkesan untuk diperolehi menggunakan pemisah makmal Mozley. Langkah pengayaan menggunakan pemisah magnetik gelean nadir bumi menunjukkan nisbah pengayaan (ER) meningkat dengan tinggi. Pada $-600+300\mu\text{m}$ pecahan bukan magnetik, nisbah pengayaan Cu adalah 2.51 dan 2.15 pada pecahan $-1180+600\mu\text{m}$. Melalui pengapungan buih balikan, perolehan Cu meningkat dan nisbah pengayaan Cu menurun dengan kenaikan saiz pecahan untuk kedua-dua keadaan pengapungan (dengan pembuih dan tanpa pembuih). Dengan demikian, kecekapan pengapungan buih meningkat pada pecahan saiz yang lebih kecil. Pada pecahan saiz $-75\mu\text{m}$ size fraction, perolehan Cu adalah 84.66% (ER 3.03) didalam sifat semulajadi hindar air (tanpa pembuih). Sementara itu dengan penambahan pembuih, perolehan Cu adalah 82.16% (ER 3.37). Terdapat peningkatan dalam nisbah pengayaan, tetapi peratusan perolehan semakin rendah dengan penambahan pembuih. Secara keseluruhan, pendekatan pemisahan fizikal mempunyai kecekapan tinggi, mudah dijalankan dan pada yang sama dapat perolehan logam dan bukan logam. Diharapkan proses pemisahan fizikal dapat dikembangkan untuk meningkatkan perolehan semula logam dari papan litar bercetak (PCBs) ini.

A Study on Physical Separation Processes for Recovery Metals from Waste Printed Circuit Boards (PCBs)

ABSTRACT

In view of increasing the waste PCBs, a physical separation process has been carried out to recover metals from waste PCBs. This research is aimed to implement an effective and environmental friendly recovery particularly copper (Cu) of waste PCBs. The physical separation process begins with comminution to produce controlled particle size. Then, the separation process was divided into two parts according optimum efficiencies at specific size range. The size fraction $-600+300\mu\text{m}$ and $-1180+600\mu\text{m}$ were separated by gravity separation using Mozley laboratory separator. Afterwards, an enrichment step of concentrate fraction was done by magnetic separation using rare-earth roll magnetic separator. Meanwhile, the size fraction $-150\mu\text{m}$, $-300+150\mu\text{m}$, and $-600+300\mu\text{m}$ were separated to froth flotation using Denver D-12 laboratory flotation cell. Characterisations of waste PCBs were performed by micrographic analysis and elemental analysis. A qualitative micrographic analysis was conducted using stereo-zoom microscope, optical microscope, and scanning electron microscopy. An elemental analysis was conducted using atomic absorption spectroscopy (AAS) analysis and energy dispersive spectrometer (EDS). Regarding on the particle size analysis, maximising recovery of physical separation is done by targeting recovery in a controlled four size range fraction; $-150\mu\text{m}$, $-150+300\mu\text{m}$, $-300+600\mu\text{m}$ and $-600+1180\mu\text{m}$. A qualitative liberation assessment of the waste PCBs particle was establish unliberated particles still remain in the waste PCBs fines ($-75\mu\text{m}$). As the highest metal element in waste PCBs, copper (Cu) recovery (R) and enrichment ratio (ER) was discussed with more emphasis in this project. By Mozley laboratory separator, Cu recovery increase from 80.85% (ER 2.07) at $-600+300\mu\text{m}$ size fraction to 89.65% (1.93) at $-1180+600\mu\text{m}$ size. Thus, the efficiency of gravity separation increases with increasing particle size. A significant of the low recovery at finer size fraction implies valuable metal loss at this size range, thereby be evidence that it is not very effective for finer particles ($-300\mu\text{m}$) was recovered by Mozley laboratory separator. For enrichment step using Rare-earth roll magnetic separator showed the enrichment ratio (ER) was highly improved. At $-600+300\mu\text{m}$ non-magnetic fraction, Cu enrichment ratio is 2.51 and 2.15 at $-1180+600\mu\text{m}$ size fraction. Through the reverse froth flotation, higher Cu recovery (R) and lower Cu enrichment ratio (ER) are noticeably with increasing particle size fraction for both flotation conditions (with and without frother). Thus, the efficiency of froth flotation is higher at finer size fraction. At $-75\mu\text{m}$ size fraction, Cu recovery is 84.66% (ER 3.03) under natural hydrophobic responds (without frother). Meanwhile with frother addition, Cu recovery is 82.16% (ER 3.37). In view of frother addition, there is improved in enrichment ratio but poor recovery percentage. Overall, the approach physical separation has high efficiency, easy to run and at same could recover metals and non-metals. It is expected that physical separation process will be developed for the upgrading of metals recovery in waste PCBs.

CHAPTER 1

INTRODUCTION

1.1 Introduction

In 2000, the numbers of personal computers consumables in Malaysia are 2.2 million and the number increase to 5.7 million in 2005 and the predict amounts, it will be increase to 11.5 Million in 2010 (Unit Perancangan Ekonomi, 2006). In view of that, the waste PCBs as a core component in computers is also increasing sharply. Considering of the environmental problem, many countries have to improve the recycle and recovery of waste PCBs rather than simply disposing it in landfill sites. According to Veit et al. (2006) the recycling of waste PCBs is a big challenge since contained plenty of toxic substances, such as brominated flame retardants (BFR), glass fibre, PVC plastic and heavy metals.

In order to develop and implement an effective and environmental friendly recovery process, physical separation techniques have been carried out necessary to recover metals from waste PCBs. Physical processing intends to recycle PCBs without consuming a great amount of energy (pyrometallurgical methods) and without generating a great amount of

effluents (hydrometallurgical methods). Physical processing also has high efficiency, easier operability and at the same time could recover non-metal materials in waste PCBs.

1.2 Physical Separation Processes

Physical separation process is drawing more attention by the researchers in recent year (Wills, 2006). Many studies have been conducted using physical processing for recovery the metallic components from waste PCBs (Eswaraiah et al., 2008; Wen et al., 2005; Yoo et al., 2009). Among other merits, physical processing is recognized as the most environmental friendly route for waste PCBs processing (Mou et al., 2004; Ogunniyi et al., 2009). In developing physical separation stream, it is important to understand the physical characteristics of waste PCBs. This is due the particles movement during separation process are based on their different properties such as size, density, shape, specific gravity and magnetic susceptibility.

According to Mou et al. (2004), a physical recycling process usually includes four stages: pretreatment, crushing, separation and ultimate refinement/reusing. The pretreatment stage includes classifying PCBs and dismantling reusable and toxic components of PCBs. The crushing stage is a key step of the physical process. In physical separation process, particle size has a great effect on the separation performance. Material properties and chosen separator process gives the limits for the separation efficiency. Some

valuable materials are lost to the tailings and some non-valuables may be misplaced in concentrate. These inefficiencies cannot be avoided. However it is possible to minimize the inefficiencies.

1.3 Problem Statement

In order to develop and implement an effective and environmental friendly recovery process, wet method of physical separation techniques have been carried out necessary to recover metals from waste PCBs. The wet method flow of physical processing intends to recovery waste PCBs without consuming a great amount of energy, effluents and solved the problem of secondary pollution from the dust created during the separation process. As expected, each separation gives optimum efficiencies at specific size range. A wet method of physical separation was employed to separate $-600+300\mu\text{m}$ and $-1180+600\mu\text{m}$ size fraction of waste PCBs using Mozley laboratory separator. Afterwards, an enrichment of concentrate fraction was done by magnetic separation using rare-earth roll magnetic separator.

A major challenge to the separation process of waste PCBs is the poor recovery of the waste PCBs fines. Fine recovery is essentially less efficient than the recovery of coarser material. Zhao et al. (2004) demonstrated the application of a type of column air separator compared to an electrostatic separator. For the $-75\mu\text{m}$ fraction, Cu recovery of 27.83% was obtained from the pneumatic separator while separation from the electrostatic at this size

was declared poor and not reported. The low recovery implies valuable metal loss at this size range, thereby complicating the finishing recovery processes (Zhang & Forsberg, 1999). An addition, these particles have poor settling characteristics in tabling (Mozley laboratory separator). The ultra fine plastic particles tend to adhere together forming clusters and entrapping small metal particles. In this project, the fine size fraction $-150\mu\text{m}$, $-300+150\mu\text{m}$, and $-600+300\mu\text{m}$ were subjected to froth flotation using Denver D-12 laboratory flotation cell. Froth flotation exploits distinct surface property of individual particles and it appears very promising for detailed investigation. Besides, there has a lack of effort to conduct empirical research into its applicability to recover metals from waste PCBs. Outstanding features of froth flotation are its selectivity, flexibility, throughput and handling of relatively fine sizes. This makes it relevant to this application.

1.4 Objectives

The objectives of this research:-

- i. To investigated the composition and particle size distribution of waste PCBs.
- ii. To observe the qualitative liberation assessment of valuable metal in waste PCBs.
- iii. To recovery valuable metals from waste PCBs by physical separation process in relation of particle size range.
- iv. To implement an effective and environmental friendly separation process to recover metals from waste PCBs.

1.5 Scope of Research

Recovery of metals from waste PCBs using a physical separation technique was proposed in this research. The research is aimed at providing effective and environmental friendly separation on recovery of metals from waste PCBs at $-150\mu\text{m}$, $-300+150\mu\text{m}$, $-600+300\mu\text{m}$ and $-1180+600\mu\text{m}$ size fraction. The approach of research consists of four phases, which are comminution and particle size analysis, gravity separation using Mozley laboratory separator, magnetic separation using Rare-earth Magnetic separator and froth flotation using D-12 Denver flotation cell. As expected, each separation method gives optimum efficiencies at specific size range.

To assemble the criteria of liberation particle size, all electronic components were initially removed from the boards. Comminution was carried out by cutting and shearing action to produce controlled particle size. Comminution or size reduction is necessary to unlock or liberated valuable metals from the gangue before separation can be undertaken. Particle size analysis by test sieving method was used to determine the optimum percentage weight distribution size by size fraction. The goal of comminution is to reach wanted particle size and liberation of valuable metals. Most separation processes would perform optimally with uniformly sized feed as it is important to limit at least the size range of the material.

From particle size analysis, a waste PCBs fine was distributed into four size

fraction: $-150\mu\text{m}$, $-300+150\mu\text{m}$, $-600+300\mu\text{m}$ and $-1180+600\mu\text{m}$. Separation and recovery of metals from waste PCBs fines was divided into two separation method regarding of size fraction. The size fraction $-600+300\mu\text{m}$ and $-1180+600\mu\text{m}$ were subjected to gravity separation using Mozley laboratory separator. Since the specific gravity differences between metals (2.6 to 19.3) and non-metals (0.1 to 1.8) of waste PCBs are great, it is favourable to take specific gravity based separation with the lack of chemicals and excessive heating requirements means it is generally environment friendly.

Afterwards, an enrichment of concentrate fraction was done by magnetic separation using rare-earth roll magnetic separator. The use of rare-earth roll magnetic separator was capable to obtain fraction with relatively high concentration of magnetic and non-magnetic fraction. Although the amount of magnetic metals (Fe and Ni) present in waste PCBs is small, it is interesting to separate it, in order to obtain non-magnetic fraction with higher copper content.

The fine size fraction $-150\mu\text{m}$, $-300+150\mu\text{m}$, and $-600+300\mu\text{m}$ were subjected to froth flotation using Denver D-12 laboratory flotation cell. This separation was done by reverse flotation using Denver D-12 Laboratory Flotation machine. In view of the fact that plastic particle of waste PCBs fines is inherent hydrophobic and it has tendency to float due to its non-wetting characteristics. Therefore, it is possible to separate mixed liberated waste PCBs fines particles into plastic fraction (hydrophobic) and heavy metallic fraction (hydrophilic) as plastics and metals have sufficient differences in floatability.

Characterisations of waste PCBs fines were performed by micrographic analysis and assay analysis. Qualitative micrographic assessment was performed to identify the liberation of surface section and distribution of metals particle for each size fraction using stereo-zoom microscope, optical microscope and scanning electron microscopy. Assay analysis was conducted using atomic absorption spectroscopy (AAS) to determine element and concentration of metals for each separation process. In terms of the major metallic element in waste PCBs, copper (Cu) element will discussed with more emphasis in this project. The result of the separability and separation efficiency is expressed in recovery percentage and enrichment ratio of the concentrate fraction. This study is expected to provide useful data for the efficient physical separation of metals from waste PCBs.

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CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

The number of personal computers discarded globally has been increasing continuously year by year. Consequently, printed circuit boards (PCBs) are the typical and fundamental component for all computers is also increasing sharply (Hall & Williams, 2007). Many studies have been carried out with regard to the recycling of waste PCBs with a view to recovering resources and at the same time preventing environmental contamination (Cui & Forsberg, 2003; Eswaraiah, Kavitha, Vidyasagar, & Narayanan, 2008; Mou, Wa, Xiang, Gao, & Duan, 2004; Takanori Hino et al., 2009; Yoo, Jeong, Yoo, Lee, & Kim, 2009). According to Veit et al. (2006) and W. J. Hall et al. (2007), the recycling of waste PCBs has now been recognized as a big challenge due consisted of a heterogeneous mixture of organic and metallic chemicals that contained plenty of toxic substances, such as brominated flame retardants (BFR), glass fibre, polyvinyl chloride (PVC) plastic and heavy metals.

The need for processing these wastes to extract the metal values and remove the non-metallic constituents has been felt all over the world. Waste PCBs, can cause serious environmental problems if not properly dispose (Chancerel & Rotter, 2009; Li, Shrivastava, Gao, & Zhang, 2004). If they are discarded randomly in the opening or land filled simply, the leachate may infiltrate into groundwater and soil. Uncontrollable incineration of waste PCBs also produces potentially hazardous by-products (including mainly dioxins, furans, polybrominated organic pollutants and polycyclic aromatic hydrocarbons) caused by burning brominated flame retardants (BFR), epoxy resins and plastics (Hall & Williams, 2007; Sepúlveda et al., 2009). The materials containing brominated flame retardants (BFR) are precursors to polybrominated dibenzo-*p*-dioxins and dibenzofurans (PBDD/Fs). These are classified as persistent organic pollutants (POPs) under the Stockholm Convention, a global treaty drawn up to protect human health and the environment. Growing attention has been given to hazardous components in waste PCBs, which pose a severe threat to human health and the sustainable economic growth as well (Huang, Guo, & Xu, 2009).

H.M. Veit et al. (2005) studied the waste management system in Europe. The generation of wastes from electrical and electronic equipment is around 7kg per capita per year in Europe. Meanwhile in the USA, a recent study predicted that over 315 million computers would be at the end of their life by the year 2004 they affirm that wastes from electrical and electronic equipment are the fastest growing waste category. This finding emphasizes the need for efficient recycling strategies. (H. M. Veit et al., 2005).