M.Tech. Dissertation Report

on

Resources Recovery from Electronic Waste

Submitted in the partial fulfilment of the requirement of the degree of

Master of Technology
In
Environmental Science and Engineering

Submitted by

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Under the supervision of

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And

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INDIAN INSTITUTE OF TECHNOLOGY BOMBAY

December 2014

Approval Sheet

This M.Tech. Dissertation titled "Resources Recovery from Electronic Waste" submitted by Mohit Arora (Roll No.123180003) is approved for degree of Master of Technology in Environmental Science and Engineering at the Centre for Environmental Science and Engineering, Indian Institute of Technology Bombay.

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This is to certify that Mohit Arora (Roll No: 123180003) was admitted to the candidacy of M.Tech. Programme on December 5th, 2014, after completing all the required courses. The details of the courses are given below:-

Course No.	Course Name	Course Credits
ES 631	Environmental Chemistry	6.00
ES 633	Environmental Microbiology and Ecology	6.00
ES 635	Air Pollution Science and Engineering	6.00
ES 637	Municipal Water and Waste Water Systems	6.00
ES 639	Physico-Chemical Treatment Technologies	6.00
ES 647	Municipal Solid and Biomedical Waste Management	6.00
ES 651	Environmental Monitoring Laboratory	6.00
ES 694	Seminar	6.00
ES 616	Energy Conversion and Environment	4.00
ES 664	Environmental Systems Modelling	6.00
ES 666	Biological Treatment Technologies	6.00
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ES 672	Air Pollution Control Technologies	6.00
ES 684	Design of Water and Wastewater Systems	6.00
HS 699	Communication and Presentation Skills	PP/NP
ES 653	Environmental Impact Assessment	6.00
MGT 647	Innovation and Entrepreneurship	6.00

IIT Bombay

Date: December 5th, 2014 Dy. Registrar (Academic)

Acknowledgements

I would like to express my deep sense of gratitude, utmost indebtedness and heartfelt thanks to my supervisor **Professor Anil Kumar Dikshit** for his valuable guidance, profound suggestions, constant backing, prolific encouragement and advice throughout the research work. I sincerely thank my co-supervisor **Professor Raj Kumar Pathak** from Thadomal Sahani Engineering College, Mumbai, for his support and continuous motivation.

My sincere acknowledgement to **IIT Bombay** for providing required facilities to perform this study, to **Ministry of Human Resource Development** for proving scholarship throughout my Masters study and sincere gratitude with thanks to **CESE staff** for their support at different ventures of this study.

There are many wonderfully supportive people who helped me throughout this project with technical as well as logistical inputs. Firstly my greatest regards for **Mr. Amit Shinde, Department of Chemical Engineering**, who helped me with manual cutting of sample and allowed to use Ball Mill for sample processing. Special thanks to him for helping me in getting printed circuit boards for my study from across IIT Bombay campus.

Mr. Karan Thakkar, Founder & MD, EcoCentric Management Private Limited helped me to get practical insight of e-waste recycling industry in Mumbai. His support by providing initial PCB sample and study material is highly acknowledged.

My sincere thanks to **Mr. DB Prabhu** and **Mr. Sujit Kochrekar**, Respose Waste Management & Research Pvt. Ltd., Mumbai for their timely support in preprocessing PCB sample. Without their support, sample preparation would have been extremely difficult. Sincere thanks to my lab mates **Shabiimam M.A.**, **Nagabhushan B.**, **Amar Shinde**, **Ashutosh Kumar** and **Joginder Singh Yadav** for various inputs and helps.

Significant part of this research was metal analysis. It was impossible to complete this work without special permissions and priorities provided by **Mr. L. S. Mombasawala**, Senior Research Engineer at Sophisticated Analytical Instrument Facility, IIT Bombay. He was kind enough to allow me for work at odd hours in analyzing more than 1100 samples. Equal acknowledgment is due for **Ms. Vinita Shetty** and **Ms. Reshma Shinde** of ICP-AES lab. Without their support, the sample analysis would have become impossible. Both of them helped in arranging slots for analysis as and when needed, many a time at very short notice.

Nothing can deny the fact that the valuable advice and encouragement by all my friends including **Rohit Saini**, **Sumit Jha**, **Lakshmideep**, **Dhruv Choudhary**, **Vikas Verma** played a crucial role in overall wellbeing at IIT Bombay. Gratitude to **Newton**, **Kamal** and **Indrakant Jha** for always being there to care and support.

I respectfully bow to my family for giving me food and education while sleeping themselves without food. Thank you for being hopeful that your sacrifices will not go in vain.

Mohit Arora

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Abbreviations

AAS	--Atomic Absorption Spectroscopy
BFR	--Brominated Flame Retardants
CFC	--Chloro Fluoro Carbons
CRT	--Cathode Ray Tube
CT	--Computed Tomography
DVD	--Digital Video Disc
EEE	--Electrical and Electronic Equipment
ESM	--Electronic Scrap Material
HCFC	--Hydro Chloro Fluoro Carbon
HFC	--Hydro Fluoro Carbon
ICP-AES	--Inductively Coupled Plasma-Atomic Emission Spectroscopy
LCD	--Liquid Crystal Display
LED	--Light-Emitting Diode
PCB	--Printed Circuit Boards
PCDD	--Poly Chlorinated Dibenzodioxins
VCD	--Video Compact Disc
VCR	--Video Cassette Recorder
VECS	--Vertical Eddy-Current Separator
WEEE	--Waste Electrical and Electronic Equipment

Abstract

Printed Circuit Boards in electronic scrap are richest source of base and precious metals, promoting economic drive for metal recovery. Recovery of these metals is a difficult exercise due to complex nature of electronic waste. India is one of the leading electronic waste processing hubs where more than 95% recyclers are unorganised/nonformal, lacking such a recovery facility. An efficient metal recovery protocol with lesser environmental impact remains unavailable to unorganised recyclers. In current study, an attempt has been made to combine various hydrometallurgical methods to achieve efficient metal recovery from Printed Circuit Boards. Mimicking mineral processing protocols for metal recovery from electronic waste has been a key feature of presented research. Printed Circuit Boards of personal computers were reduced to a size ≤ 2.5 mm and were used as sample for metal recovery. Copper and silver were extracted by selective leaching using Nitric Acid. Maximum extraction efficiency of 96.8% and 99.9% were achieved for copper and silver respectively. Nitric Acid was extracted using solvent extraction method to minimize the environmental damage of remaining waste as well as for reuse in next leaching cycle. Electrodeposition of copper was done on copper cathode with stainless steel and lead anodes with a maximum recovery efficiency of 89.6%. For extraction and recovery of gold and silver, various techniques used in mineral processing were applied in this research. Thiosulfate leaching was chosen for gold extraction due to its proven environment friendly properties. Maximum gold extraction efficiency of 74.3% has been achieved in this work. To recover gold from pregnant solution, zinc cementation approach was used. Up to 85.9% gold was recovered with zinc cementation. Success of thiosulfate leaching and cementation confirms the validity of mineral processing techniques in metal recovery from electronic waste. Research in this study can serve as a backbone for potential environmentally sound technology towards efficient metal recovery from electronic waste for small and medium scale recyclers.

Keywords: Electronic Waste, Printed Circuit Boards, Metal Recovery, Hydrometallurgy, Thiosulfate Leaching, Copper Electrowinning, Cementation

Chapter 1
Introduction

1.1 Background

Information and Communication Technology has been the most distinguished achievement of 21st century. Additionally, this era has seen immense success towards technological advancement in pursuit of achieving complete automation. Electronics remains the heart of Information Technology as well as automation. Rapid technological innovations in last decades have increased the variants of each service and product. Electrical and electronic equipment (EEE) have been one of the major areas of technological advancements and so the fastest growing manufacturing sector across the globe. Interestingly, for countries, the race for development may seem achieving higher Gross Domestic Products and better economy but for citizens the only sign of development remains improved lifestyle. In this world today, it may not be wrong to say that the major contribution in improved lifestyles remains electrical and electronic products. So, increasing world population led to the consumer oriented growth and increased demand for electrical and electronic equipments. Continuous technological advances lead to the consumer's encouragement for frequent upgrading and purchase of electrical and electronic products. Perhaps development or improved lifestyle as we may call for people, takes its own price and so the globe has to pay. Improved as never before yet fascinating life style and countless technological breakthroughs' have created novel environmental threats. Increased demand based purchase along with relatively short lifetime of electrical and electronic equipment has resulted into huge electronic waste generation. Increasing generation of waste electrical and electronic equipment (WEEE) has posed major environmental concern for its disposal and management. Electronic waste is an emerging environmental threat because of its toxic and hazardous nature. Currently, major fate of electronic waste is reuse, recycling, incineration and land filling (Wath et al., 2011).

In addition to its toxic nature, Electronic waste is a valuable business opportunity due to significant amount of base and precious materials. Recovery of resources from electronic waste, thus remains a logical approach to deal with it.

The world's ever increasing consumption of electronic and electrical goods has created a corresponding explosion in electronic scrap containing toxic chemicals and heavy metals. E-wastes contain over a thousand different substances many of which are toxic and potentially hazardous to the environment and human health, if not handled in environmentally sound manner. E-waste is used as a generic term embracing various types of waste containing, principally electrical and electronic components.

1.2 Electronic Waste

According to definition given in e-waste management guidelines (2011) provided by Government of India: “E-waste comprises of wastes generated from used electronic devices and households’ appliances which are not fit for their original intended use and are destined for recovery, recycling or disposal. Such wastes encompass wide range of electrical electronic devices such as computers, hand held cellular phones, personal stereos, including large household appliances such as refrigerators, air conditioners etc.” Whereas Electrical and electronic equipment has been defined as “equipment which is dependent on electrical currents or electro-magnetic fields to be fully functional” (MoEF, 2011).

Internationally, the most accepted definition of e-waste is the definition as per the WEEE Directive which defines electronic waste, "e-waste" or "Waste Electrical and Electronic Equipment" ("WEEE") as the “waste material consisting of any broken or unwanted electrical or electronic appliances including all components, subassemblies and consumables which are part of the product at the time of discarding” (URL: 01).

1.2.1 Classification of E-waste

Composition of e-waste is very diverse and differs in products across different categories. It contains more than 1000 different substances, which fall under “hazardous” and “non-hazardous” categories (Wath et al., 2011). The presence of elements like lead, mercury, arsenic, cadmium, selenium, and hexavalent chromium and flame retardants beyond threshold quantities in e-waste classifies them as hazardous waste. The Directive of the Parliament and European Union Council on waste electrical and electronic equipment subdivides WEEE into ten different categories (EU, 2002).

1. **Large Household Appliances:** Refrigerators/freezers, Dryers, Air conditioners, Washing machines, Dishwashers etc.

2. **Small Household Appliances:** Toasters, Coffee makers, Irons, Hairdryers, Vacuum cleaners etc.
3. **Information Technology and Telecommunications Equipment**: Personal Computers, Telephones, Mobile phones, Laptops, Printers, Scanners, Photocopiers etc.
4. **Entertainment and Consumer Equipment:** Televisions, VCR/DVD/VCD Players, Stereo Equipment, Electric Toothbrushes, Transistor Radios etc.
5. **Lighting Equipment:** Fluorescent Lamps, Sodium Lamps etc.
6. **Electrical and Electronic Tools:** Handheld Drills, Electric Saws, Screwdrivers, Lawn Movers etc.
7. **Toys, Leisure, Sports and Recreational Equipment:** Coin slot machines, Tread mills, Electric train sets, PlayStation, Gameboy etc.
8. **Medical Equipment Systems:** CT Scanners, X-ray Machines etc. (with the exception of all implanted and infected products)
9. **Monitoring and Control Instruments:** Smoke Detector, Heating Regulators, Thermostats, Measuring, Weighing or Adjusting Appliances etc.
10. **Automatic Dispensers:** Bank ATMs, Issuing machines, Automatic Dispensers for Hot Drinks, Automatic Dispensers for Hot or Cold Bottles or Cans etc.

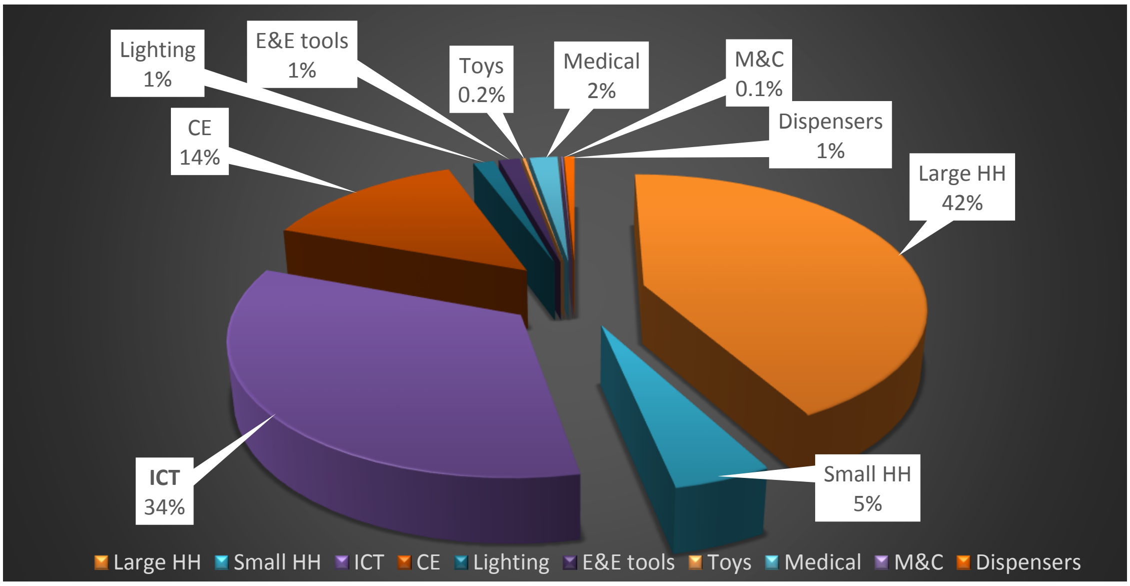

Figure 1.1 E-waste Distribution (Widmer et al., 2005)

Of the ten categories listed above, Categories 1–4 account for almost 95% of the WEEE generated as shown in figure 1.1 (Widmer et al., 2005).

1.2.2 Building Blocks of E-waste

Electrical and electronic equipment's consist of a combination of several basic building blocks. Each of E-waste items has been classified with respect to twenty six common components, which could be found in them. These components form the "Building Blocks" of each item and therefore they are readily "identifiable" and "removable". These components are metal, motor/compressor, cooling, plastic, insulation, glass, LCD, rubber, wiring/ electrical, concrete, transformer, magnetron, textile, circuit board, fluorescent lamp, incandescent lamp, heating element, thermostat, BFR-containing plastic, batteries, CFC/HCFC/HFC/HC, external electric cables, refractory ceramic fibres, radioactive substances and electrolyte capacitors.

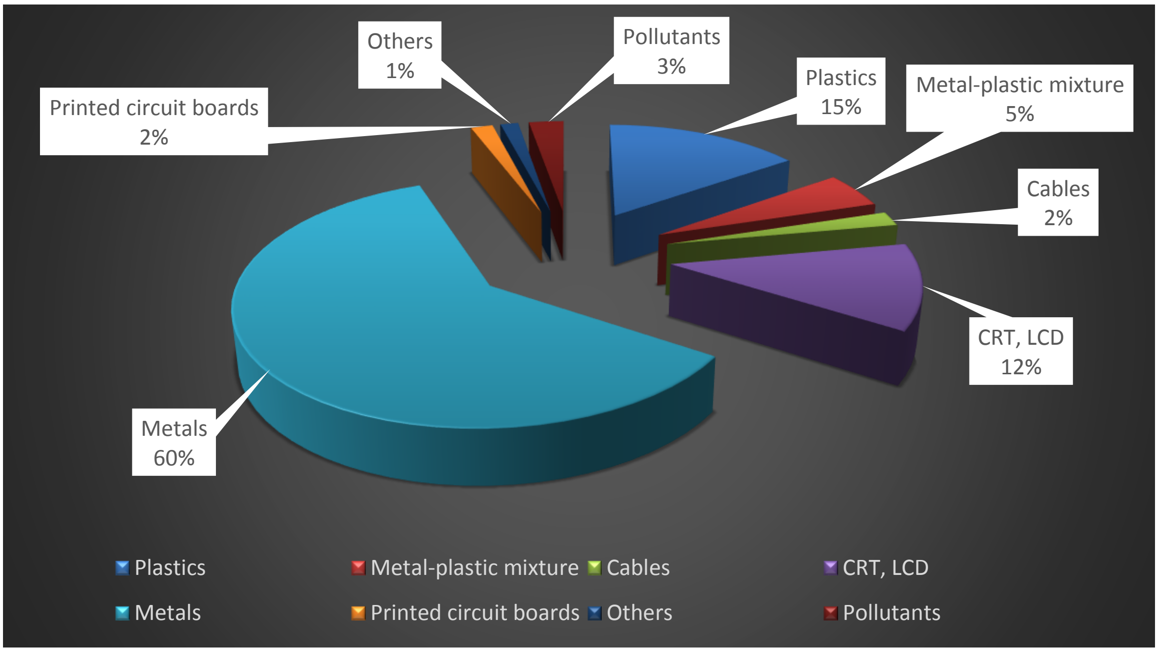

Figure 1.2 Typical Composition of E-waste (Widmer et al., 2005)

Some of the specified building blocks are (EMPA, 2004):

- Printed Circuit Boards/Assemblies
- Plastic Containing Flame Retardants

- Cables, cords, wires, and connectors
- Mercury switches and breakers
- Display equipment such as cathode ray tubes and liquid crystal display (LCD)
- Sensors and accumulators
- Data storage media
- Light generating devices
- Capacitors, resistors and relays
- Batteries

1.2.3 E-waste Production Rate

WEEE is the fastest growing waste stream in the world with a 3-5% increasing rate per year then municipal wastes (UNEP, 2009). According to estimates by the United Nations Environment Programme, around the world about 50 million tons of e-waste is produced annually, of which only 10% is recycled. Each European inhabitant produces about 20 kg each year (Delfini et al., 2011). Millions of tons of electronic waste from obsolete computers and other electronics are being generated in the U.S. each year. In 2003, more than 54 million PCs were sold in United States. The rate of PC obsolescence now exceeds the rate of production. The lifespan of PCs is getting shorter as a result of technology development and higher standards for PCs. The International Association of Electronics Recyclers (IAER) estimated that in 2003 the U.S. electronics recycling industry consisted of just over 7000 employees and annual revenue of over US$ 700 million (Kang and Schoenung, 2005).

It is observed in recent years that large volume of e-waste is being exported from western countries to Asian countries like China, India for disposal. Recycling business in western countries is becoming economically non-viable due to rising cost of manpower and availability of input materials for running the plant in full capacity. The western countries are, therefore, compelled to find out alternative destinations for disposal, where the labour cost is comparatively low and the environmental laws are not enforced so strictly. The imported materials are thus reaching through illegal routes in India, China for recycling at small-scale units in non-formal sector. These units use primitive, non-scientific, and non-environment-friendly methods. It is estimated that 95% of the e-waste recycling in India has been carried out in non-formal units, Moreover, apart from the domestic generation of e-waste; the imported electronics waste has also contributed a significant impact in the total inventory of the material. India is becoming a big market for imported e-waste. A study indicates that PCs imported to Delhi in 2003 was nearly 3,600,000 Kg/year. Another study predicts that the nearly 50,000 to 70,000 tons of e-waste is being imported annually to India. Most developed countries, find it financially profitable to send e-waste for re-use/recycling in developing countries. The cost of recycling of a single computer in the United States is $20 while the same could be recycled in India for only US $2, a gross saving of US $18 if the computer is exported to India. It is estimated that more than 2000 non-formal recyclers are involved in the recycling business, Over 10,000 numbers of unskilled workers are involved in non-formal sector alone in Delhi (Chatterjee, 2012).

Table 1.1 shows the quantitative estimation of electronic waste produced in India with recycling. The 2.2 million computers had become obsolete in 2007. India had about 20 million computers in 2007. Around 14 million mobile handsets had been replaced in 2007 (Chatterjee and Kumar, 2009). According to Centre for entrepreneurship development, Government of Gujarat, E-waste volume is expected to increase at a rate of 300% per annum in developing countries. In India, Currently installed and functioning capacity in the organized sector for electronic waste recycling is only about 100,000 tons while the generation rate of e-waste is about 800,000 tons. The current market size itself is sufficiently large and also growing at more than 30 %. Hence there is room for many more new recyclers (URL-02).

Table 1.1 Estimation of Electronic Waste Generation in India (Chatterjee, 2012)

Waste Electrical and Electronic Equipment	**Weight (MT)**
Domestic Generation	3,32,979
Imports	50,000
Total	**3,82,979**
WEEE Available for Recycling	1,44,143
WEEE Actual Recycled	19,000

Table 1.2 shows the expected electronic waste generation compared to the electrical and electronic equipment coming to market in metric ton per year. The Central Pollution Control Board, India, estimated 0.1347 million MT of e-waste was generated in the country in the year 2005 (Chatterjee, 2012).

Table 1.2 Electronic Appliance and E-waste Generation Rate, India (UNEP, 2009)

Electrical and Electronic Equipment	Quantity of EEE Put on Market (Metric Tons/Year)	E-Waste Generation (Metric Tons/Year)
Personal computers	1,40,800	56,300
Printers	12,000	4,700
Mobile phones	9,300	1,700
TVs	4,50,000	2,75,000
Refrigerator	2,11,500	1,01,300

1.2.4 Resources in E-waste

Electrical and electronic equipment are made of various resources, which can be recovered after equipment becomes obsolete. Table 1.3 shows the distribution of metals in waste electrical and electronic equipment.

Table 1.3 Metals in Different Scrap Samples (Cui and Zhang, 2008)

	Weight (%)					Weight (ppm)		
Electronic waste	**Fe**	**Cu**	**Al**	**Pb**	**Ni**	**Ag**	**Au**	**Pd**
TV board scrap	28	10	10	1	0.3	280	20	10
Mobile phone scrap	5	13	1	0.3	0.1	**1380**	**350**	**210**
Portable audio scrap	23	21	1	0.14	0.03	150	10	4
DVD player scrap	62	5	2	0.3	0.05	115	15	4
Calculator scrap	4	3	5	0.1	0.5	260	50	5
PC mainboard scrap	4.5	14.3	2.8	2.2	1.1	639	566	124
TV scrap (CRTs removed)	-	3.4	1.2	0.2	0.038	20	<10	<10
PC scrap	20	7	14	6	0.85	189	16	3
Typical electronic scrap	8	20	2	2	2	2000	1000	50
Printed circuit boards	5.3	**26.8**	1.9	-	0.47	**3300**	**80**	-

E-waste contains more than 1000 different substances, many of which are toxic, such as lead, mercury, arsenic, cadmium, selenium, hexavalent chromium, Polychlorinated Biphenyls Brominated Flame Retardants, Dibenzodioxins and Dibenzofurans. About 70% of the heavy metals (mercury and cadmium) in US landfills come from electronic waste. Consumer electronics make up 40% of the lead in landfills. These toxins can cause brain damage, allergic reactions and cancer (IMRB International, 2010).

1.3 Research Problem

India is one of the major e-waste producer among developing countries due to increasing penetration of electrical and electronic products. Additionally, India remains a major hub for receiving global e-waste export. There are lot of opportunities for converting the threat of e-waste into a profitable business because of its resource rich nature. In India, most of electronic waste first goes into reuse by local refurbishing markets. Parts which are unusable goes for resources recovery in its second step of e-waste processing.

In India, resources recovery form electronic waste is governed by formal and informal sectors while more than 95% of e-waste is handled by informal sector (Banerjee, 2012). Formal (organized) sector is following conventional methods of resources recovery with little advancement through mechanical processing while informal sector is fully dependent on manual dismantling along with acid bath for metal recovery. Manual and conventional resources recovery methods pose great health and safety risk. The efficiency of resources recovery also remains very low leaving behind remains to landfill or dumping site creating much bigger environmental risk. An important aspect of waste management is waste characteristics. Electrical and electronics equipment composition is very diverse and complex so the resultant WEEE also shows varied complexity. For effective recycling and recovery process, optimized technological processes based on e-waste characterization are of utmost importance. Currently, only few e-waste recyclers dictate the global recycling due to very high capital cost involved in sophisticated recovery facility for complex electronic waste like Printed Circuit Boards. Informal players and small formal recyclers sell such complex electronic waste to giant recyclers due to lack of profitable technology. Some small scale recyclers do practice metal recovery from complex e waste but such techniques remains highly unscientific and environmentally degrading. Extensive research is required for enhancing the efficiency of resources recovery in safer ways of complex e-waste processing, minimizing health-safety and environmental concerns.

1.4 Motivation of the Study

For emerging economies like India, it is mandatory to follow the concept of circular economy to ensure maximum utilisation of materials. While one may say that there is a need of formal legislative system and guidelines to become a circular economy, Indian markets shows inherent characteristics of being in circular framework through rigorous reuse applications. Material inflow through waste import and electronic waste generation satisfies the demand for cheap second-hand electrical and electronic equipment. There is a stabilised system in developing countries like India for trading, repairing and recovering materials from electronic waste. Main reason for emergence of such a system being presence of precious metals like Gold (Au), Silver (Ag), Platinum (Pt), Palladium (Pd) and base metals like Copper (Cu), Aluminium (Al), Iron (Fe), Nickel (Ni) in high amount compared to very low (~ 2% by weight) toxic elements in electronic scrap (Böni and Widmer, 2007). Tay et al. (2013) reported that the gold content in electronic scrap material is significantly higher compared with natural gold ores, creating an economic drive and excellent reason for the recycling of electronic waste. While system has been efficient in harvesting reuse potential, it still lacks environmentally sound technology for metal recovery from electronic scrap. This system serves as a source of livelihood for thousands of urban and rural poor but it often causes severe risks to humans and the local environment. Most of the participants in this sector are either not aware of the risks, or do not have financial capital to procure safer technology and equipment. For countries like India where informal sector remains the biggest player of electronic waste recycling, efficient recovery of metals from complex electronic waste remains a distant dream. Countries like India and China still lack an efficient technology for metal recovery from electronic waste (MoEP, 2014). Keeping in mind the need for an indigenous technology to efficiently recover metals from Printed Circuit Boards with such simplicity that both formal and informal sector can utilise it, it would be interesting to mimic the metal recovery from minerals/ores for developing a technique implementable for electronic waste. Such a technique will not only serve as an environmentally sound treatment method but also as a source of livelihood for thousands of people involved in recycling sector.

1.5 Research Objectives

This research aims at development of appropriate technological protocol for recovery of metals present in electronic waste. Objectives of this research are:

1. To perform a comprehensive literature survey for understanding the past efforts made to recover metals from electronic waste and metal ores.
2. To extract the metals especially Copper, Silver and Gold from electronic waste using hydrometallurgical approach.

3. To recover the metals from pregnant solutions.

Scope of this research is limited to metal recovery from Printed Circuit Boards of Personal Computers.

1.6 Outline of Thesis

First chapter, introduction, starts with research background, overview of electronic waste, research problem and motivation behind study along with objectives of the research. In chapter two, a complete literature review related to the research problem has been presented which includes the past efforts made by the researchers in mechanical as well as metallurgical approach of metal recovery. Chapter three describes the research methodology adopted for presented work. Chapter four includes various results and findings of the present research along with discussion. Chapter five presents the summary and conclusions drawn on the basis of various findings of current research. The scope for future work has also been highlighted in this chapter.

Chapter 2
Literature Review

As discussed earlier, treatment of electronic waste majorly involves reuse, recycling, incineration and landfilling. Reuse has been based on manual dismantling while there is ongoing search for an environmentally sound technology for recycling and metal recovery. Incineration by traditional incinerator designed for municipal solid waste pose greater danger because copper is a catalyst for dioxin formation which forms when flame-retardants are incinerated. Incineration of brominated flame retardants at low temperature poses serious health concerns. It has been reported that emissions from waste incineration account for 36 tonnes per year of mercury and 16 tonnes per year of cadmium in the European Union Countries (Cui and Zhang, 2008). Landfill remains the most favoured destination for e-waste in many countries even after legislations in place banning such practice keeping in mind the hazardous nature of waste and subsequent environmental damage (URL-03).

Significant research has been done to explore the possibilities of metal extraction and recovery from electronic waste. Cui and Forssberg (2003) described three broad steps for electronic waste recycling. First step involves disassembly to separate hazardous or valuable parts for specific treatment. Second step involves upgrading by mechanical or metallurgical processing to extract the desired resources and third step is to refine the recovered materials by retreatment or purification applying chemical and/or metallurgical process. Refined material should potentially be pure or may have direct usability.

While disassembly majorly remains manual or mechanical, upgrading has been dominated by mechanical and metallurgical approaches. Majority of research under metallurgical processing has been based on understanding of metal recovery from mineral ores in mines. Second step of recycling process remains the decisive step for a better technological protocol of metal recovery which has been discusses in next section.

2.1 Mechanical Processing

Mechanical processes, such as screening, shape separation, magnetic separation, Eddy current separation, electrostatic separation, and jigging have been widely utilized in recycling industry. However, for maximum separation of materials, WEEE should be shredded to fine particle size range, generally below 5 or 10 mm (Cui and Forssberg, 2003).

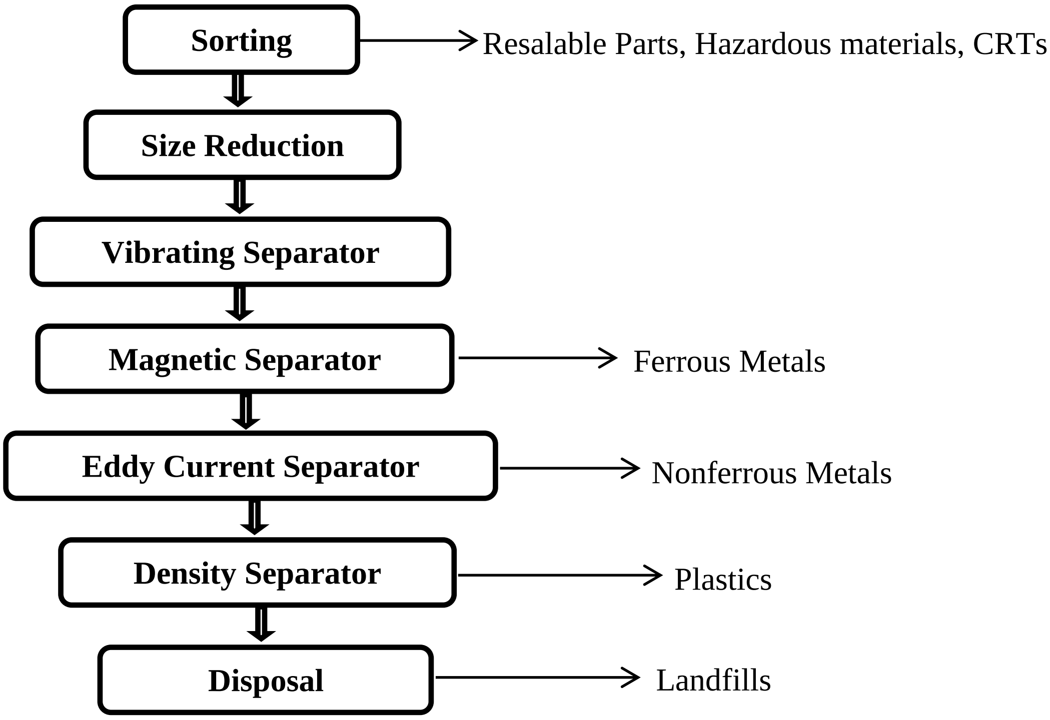

Figure 2.1 Schematic Diagram of Mechanical Recycling Process of WEEE (Kang and Schoenung, 2005)

2.1.1 Disassembly

Selective disassembly or dismantling has been an indispensable step in waste recycling because of reuse priority, removal of selective hazardous components and separation of precious/desirable parts such as printed circuit boards, cables, and plastics. It is also crucial for simplification of subsequent steps in pursuit of recovery of materials. Dismantling remains a manual process with application of various tools for increased efficiency and lower time. However, it may be a costly affair to perform manual dismantling of products containing low-grade precious metals and copper like TV sets, video recorders etc. (Kopaeck and Kopaeck, 1999).

2.1.2 Screening and Shape Separation

Screening is very significant step for size uniformity as well as upgrading metal content. As the particle size-shape properties of metals differ from plastics and ceramics, this process can play a crucial role in making metal rich streams. Rotating screens have been significantly used for metal recovery. Rotating screen shows high resistance to blinding, which helps in the process as the particle shapes and sizes are highly varying in electronic waste. Another widely accepted technique is vibratory screening specifically for non-ferrous recovery sites (Cui and Forssberg, 2008). Shape separation has played an important role in controlling particle properties. Tilted plate and sieves are the most common approach while inclined conveyor and vibrating plate have also been used to recover metals from waste cable, printed circuit boards, TV and personal computers. (Gungor and Gupta, 1998).

2.1.3 Magnetic Separation

Magnetic separators have been extensively used for separation of ferromagnetic materials. Low-intensity drum separators separates ferromagnetic metals from non-ferrous/nonmagnetic materials in waste stream. With latest advancements in High-intensity magnetic separators, it is now possible to separate copper alloys from the waste matrix (Cui and Zhang, 2008).

2.1.4 Electric Conductivity based Separation

Based on different electric conductivity of materials, it is possible to separate them through Electric conductivity-based separation. Eddy current separation, Corona electrostatic separation, and Triboelectric separation are major techniques based on this concept. Eddy current separators have been highly successful in the recovery non-ferrous metals from shredded coarse size electronic scrap. Electrostatic separator with application of corona charging separates raw materials into conductive and non-conductive fractions. The extreme difference in the electric conductivity of metals and non-metals forms the basis of corona electrostatic separation electronic waste recycling. Recovery of copper, aluminium and precious metals from electronic scrap has been performed using this technique. Triboelectric separation separates plastics based on the difference in electric properties, which has been highly desirable for plastic recovery and refining (Schubert and Warlitz, 1994).

2.1.5 Density/Gravity based Separation

Heavy materials can be separated from lighter ones based on their density difference. This serves as the principle for density based separation and has been extensively applied for the separation of metals from non-metals. Gravity concentration works on the fact that materials of different specific gravity have different relative movement in response to the force of gravity and other forces like resistance by fluid (air/water). Particle motion in a fluid is affected by multiple factors including particle’s density, size as well as shape so size control of feed determines the effectiveness of gravity/density processes. Jigging is one of the widely used methods of gravity concentrations in mineral processing industry for concentrating relatively coarse materials. With a fairly uniform feed size, good separation of a narrow gravity range can be achieved, acting as an efficient way for sorting small pieces of metals. Heterogeneity and high complexity of electronic waste makes it difficult to be separated by jigging process (Sohaili *et al.*, 2012).

2.2 Metallurgical Processing

For refining purpose, recovered metals through mechanical processing, are either melted or dissolved in solutions using metallurgical techniques for purification. Refining step extensively utilise application of:

- Pyro-metallurgy
- Hydrometallurgy
- Bio-metallurgy

While pyro-metallurgical processing and hydrometallurgical processing have been intensively used for metal recovery, bio-metallurgical processing approach has also been used recently to recover metals from electronic waste based on works in mineral processing for metal recovery. Most of the hydrometallurgical oppressions to recover metals from electronic waste are influenced or adopted from mineral processing methods of ores but application of techniques from the field of mineral processing to the treatment of electronic waste has limitations. It is evident that the particle size as well as material contents are very different in mineral processing and electronic waste system. It is possible though to use these techniques with certain modifications for metal recovery from electronic waste. With optimization of mineral processing protocols, alternatives for metal recovery from e-waste can be generated.

For recovery of non-ferrous as well as precious metals from electronic waste, traditionally, Pyrometallurgical processing has been extensively used. Pyrometallurgical approach includes incineration, smelting in a plasma arc or blast furnace, drossing, sintering, melting and reactions in a gas phase at high temperatures. The process involves burning of the crushed scraps in a furnace or in a molten bath to remove plastics and the refractory oxides form a slag phase together with some metal oxides.

Pyrometallurgy has been extensively used by big recycling industries for recovery of precious metals from electronic waste but most methods have certain limitations. Cui and Zhang (2008) reported that:

- Aluminium and Iron cannot be recovered as metals by integrated smelters and are transferred to slag. Addition of aluminium influences the slag properties which are undesirable for further refining.
- Halogenated flame retardants of electronic scrap lead to the formation of dioxins. For avoiding dioxins, special installations and measures are needed.
- State-of-the-art smelters for electronic scrap processing are highly capital intensive.
- Ceramic components and glass in the electronic waste increases slag from blast furnaces increasing the loss of precious and base metals.
- Huge energy consumption and limited energy recovery options by utilizing of organic constituents as a reducing agent.
- Pyrometallurgical process allows obtaining precious metals feed only at the very end of the process.
- Pyrometallurgy can achieve only partial metal separation, necessitating application of hydrometallurgical and/or electrochemical processing for final recovery.

Keeping above limitations in mind along with requirement of huge capital investment, high feed input and sophisticated technology, this approach remains distant from small scale operations and so from the small recyclers.

Biometallurgy has been utilised in the form of bioleaching for metal extraction and biosorption for metal recovery in mineral processing. Bioleaching has been tested in few studies for potential metal recovery from electronic scrap material yet it remains at naïve state for lab scale operations (Brandl et al., 2001).

In the 1970s and early to mid-1980s, the predominant method of recycling of electronic scrap was blast furnace smelting in conjunction with secondary copper or lead smelters. Since the mid-1980s, the trend has shifted toward the hydrometallurgical processing (Sum, 1991).

Precious metal recovery remains most attractive from an economic point of view, which is majorly based on hydrometallurgical processing. Hydrometallurgical approach on the other hand remains more exact, predictable, and easily controllable compared to pyrometallurgy (Quinet et al., 2005). Keeping in mind the interest of small scale recyclers, hydrometallurgy also remains less capital intensive, easy to practice and workable at low electronic waste feed. These reasons make it a preferred choice of research.

Hydrometallurgical processing involves a series of acid or caustic leaches of electronic waste. The pregnant solution is then subjected to various procedures of separation, purification like precipitation, solvent extraction, adsorption and ion-exchange for isolation and/or concentration of the metals of interest. Concentrated solutions are the subjected to various techniques of metal recovery like electro-refining processes, chemical reduction, or crystallization (Cui and Zhang, 2008).

Various studies have been done to extract metals from PCBs. Koyama et al. (2006) demonstrated copper extraction from fine powdered PCBs (1.5- 3.4 mm size) using cupric ammonical solution in nitrogen environment emphasizing that the method has high selectivity for copper leaching and during electrowinning of copper power consumption is low due to oxidation of Cu (I) at anode. Research highlighted that the leaching rate for copper inside the PCB was much lower due to the fact that limited surface remains exposed to leaching agent. It was recommended for achieving 82% leaching of copper from PCBs, size reduction upto 1.5 mm must be done.

Sulphuric and Hydrocholric Acid solutions have been extensively used as leaching agents for metals but it was reported that the separation process becomes more complicated because of the poor selectivity of leaching agents (Koyama et al., 2006)

Birloga et al. (2013) found that presence of metallic copper adversely affects the gold extraction process. Study achieved 76% Copper extraction using sulphuric acid and hydrogen peroxide. With a particle size less than 2 mm, a gold extraction efficiency of 69% was achieved using thiourea leaching.

Oh et al. (2003) achieved 95% extraction of copper, iron, zinc, nickel and aluminium by 2 Molar Hydrochloric Acid with 0.2 M Hydrogen Peroxide at 12 hour time maintaining a temperature of 86°C. Particle size range of crushed PCB was 1 mm.

Joda and Rashchi (2012) used an empirical model to obtain optimum extraction conditions for Silver and copper using Nitric Acid. The approach was based on central composite design and response surface methodology. Optimum parameters so achieved were used to further standardise the copper and silver recovery. 87% silver and 98% copper was achieved using nitric acid leaching for extraction and silver precipitation along with copper electrowinning.

Kulandaisamy et al. (2003) reported advantages of Thiosulfate leaching for gold over cyanide and thiourea leaching due to environmental hazard associated with cyanide, interference of base metals in gold leaching by thiourea and cheaper availability as well as higher dissolution rate of gold in thiosulphate leaching.

Abdel-Aal and Farghaly (2007) reported cementation study depositing 100% silver from nitric acid rich solution with addition of Zn metal powder. Silver was leached from photographic films with 6 N nitric acid and extraction efficiency of about 98% of the silver was achieved. Particle size 2 mm, temperature 90 °C, reaction time 50 min and solid:liquid ratio 1:50 g/ml. 100% of silver was cemented with 12.5 stoichiometry of zinc powder at pH value of 3.5±0.5 for 15 min.

Yen et al. (1996) used thiosulfate leaching to extract gold from copper rich ore having concentration of 7.2-7.9 gm gold/ton of ore. 0.5 M Thiosulphate, 6 M Ammonia solution along with 0.1 Molar Cu^{+2} ion concentration achieved an extraction of 95-97% gold after 24 hour retention time.

Bas et al. (2011) achieved 62% gold recovery using 0.5 M Ammonical Thiosulphate, 0.5 M Ammonia solution along with 0.025 M Cu^{2+} ion concentration. Study concluded that efficiency is much higher that cyanide leaching and thiosulphate concentration along with ammonia concentration are determining factor for extraction efficiency.

Abbruzzese et al. (1995) reported Thiosulphate as a non-toxic and environmentally friendly alternativeleaching agent for gold. Thiosulphate leaching is carried out in the presence of ammonia (NH_3) and cupric copper (Cu^{2+}) as catalyst. Ammonia plays a crtical role in stabilizing copper (II) and to minimise the decomposition of thiosulphate through the reduction of Cu (II) to Cu (I). Equation 2.1 shows chemical reaction involved.

$$\mathbf{Au + 5S_2O_3^{2-}{}_{(aq)} + Cu(NH_3)_4^{2+} \longrightarrow Au(S_2O_3)_2^{3-}{}_{(aq)} + 4NH_{3(aq)} + Cu(S_2O_3)_3^{5-}{}_{(aq)}} \quad \textbf{(Eq 2.1)}$$

Arima et al. (2002) reported Zinc precipitation as an effective method recovery of Gold from thiosulfate solution based on cementation. Study reported that 100% gold was recovered with 30:1 mass ratio of Zinc to gold. Process is explained in Eq. 2.2

$$\mathbf{2Zn + 2Au(S_2O_3)_2^{3-} + 4\ NH_3 + 2H_2O \longrightarrow}$$

$$\mathbf{[Zn(S_2O_3)_2]^{2-} + [Zn(NH_3)_4]^{2+} + 2S_2O_3^{2-} + 2OH^- + H_2\uparrow + 2Au\downarrow} \qquad \textbf{(Eq 2.2)}$$

Above discussed studies have been used as the basis to formulate the methodology for research presented in this study.

Chapter 3
Materials and Methods

3.1 Materials

3.1.1 Glassware and Reactors

Experiments were conducted in glassware supplied by Borosil, India. Major glassware include conical flasks, beakers and Erlenmeyer flask of various volume. For sample collection glass pipettes of 10 ml and 25 ml volumes were used. Sample dilution for metal analysis were performed using micropipette supplied by Accupipet. Sampling tubes supplied by Tarson Plasticwares were used. For the purpose of Well Stirred Tank Reactor (WSTR), specially designed borosilicate reactors of 1 Litre and 2 Litre volumes were used. For copper electrowinning, electrodeposition assembly were prepared with dimensions of 8 cm x 8 cm x 15 cm using acrylic sheets.

3.1.2 Chemicals

Various chemicals purchased for this research were of Analytical Grade (A. R.). Nitric Acid (69%), Hydrochloric Acid (35%), Hydrofluoric Acid (40%), Hydrogen Peroxide (30%), Ammonium Hydroxide, Zinc powder (pure), Copper Sulphate, Tributyl Phosphate and Ammonium Thiosulphate were purchased from Merck. Boric Acid (extra pure powder) was purchased from S. D. Fine Chemicals Ltd. Mumbai, India.

3.1.3 Electronic Waste

This research is focused on Printed Circuit Boards. Printed Circuit Boards (PCB) used in this study were collected from electronic waste discard in various departments of Indian Institute of Technology Bombay. In order to ensure uniformity in the sample, Central Processing Units (CPU)/System units of Desktop computers were chosen to be the main source of PCBs.

3.1.4 Instruments

Waste Shredder and Grinder was used from Respose India Limited, Mumbai. An indigenous Ball Mill from Department of Chemical Engineering at IIT Bombay was used. Various sieves designed as per the British standard specifications for size fractionation were supplied by Jayant test sieves, Mumbai. Microwave digester model Mars6 manufactured by CEM Corporation was used for complete digestion purpose. Model ARCOS of Inductively Coupled Plasma - Atomic Emission Spectrometer was used for metal analysis which is manufactured by M/s. Spectro, Germany. For heating and mixing purpose, indigenous hot plate magnetic stirrers were used. For electrodes of electro-deposition assembly, metal sheets namely copper, lead and stainless steel were purchased and cut into desired size electrodes. An Indigenous DC current source was used to provide desired current density.

3.2 Methods

3.2.1 Sample Preparation

The CPUs were manually dismantled to take out the PCB's. Freely detachable mountings on the Printed Circuit Boards were first removed manually. PCBs were then subjected to shredding by a waste shredder from Respose India Limited, Mumbai. Resultant shredded material was in the size range of 5-7 cm. This material was first used as a feed to ball mill at Department of Chemical Engineering, IIT Bombay, as described by some studies to convert it into 1-2 mm size range. Due to the ductile material of PCBs, ball mill failed to achieve comminution. Shredded material then fed to the waste grinder at Respose India Limited, Mumbai, India which gave the output particle size ≤ 4 mm. Such granules were further classified into various size fraction using size separating sieves supplied by Jayant Sieves, Mumbai. Size fraction ≤2.5 mm was used for further experimental work.

3.2.2 Microwave Digestion

For analysing metals in pre-processed PCB material, it is important to liberate the metals into a solution because metals are randomly distributed in the sample particles. For metal liberation into a solution, highly oxidative digestive conditions are required with high temperature. For complex samples like PCBs, it is difficult to digest samples on hot plate or open heating. For effective digestion, Microwave Digester model Mars6 manufactured by CEM Corporation was used. Optimization of working conditions was performed along with various acids to achieve the complete digestion of samples. Triplicate samples, each 1 gram pre-processed material, were used for microwave digestion using Nitric Acid, Hydrogen Peroxide and Hydrofluoric Acid solution. Various combinations of acid ratio were used to standardise the complete digestion. After adding 1 gram sample to digestion tubes, Hydrogen Peroxide was added followed by Hydrofluoric Acid and Nitric Acid. A total of 14 ml acid volume was added to each solid sample. 10 minute pre-digestion was given after adding acid to reduce the fumes. Procedure was carried out in a fume hood (for safety). Microwave digestion was performed in a two stage protocol.

Table 3.1 Operating Parameters of Microwave Digester

Stage	Temperature (°C)	Power (Minutes)	Heating Time (Minutes)	Holding Time (Minutes)	Cooling Time (Minutes)
Stage 1	220	1000W	25	20	15
Stage 2	200	1000W	20	15	15

3.2.3 Initial Metal Analysis

The exact quantitative data on the metal composition of PCBs can be obtained by using atomic absorption spectroscopy (AAS) or inductively coupled plasma-atomic emission spectroscopy (ICP-AES) after digesting the sample in combination of acids. Although AAS can analyse a number of elements in the range of ppm or ppb concentration, it is more time consuming and cost intensive (Chatterjee and Kumar, 2009).

Pre-processed material was analysed to get the quantitative level of metals mainly Copper, Silver, Gold, Platinum and Palladium using inductively coupled plasma-atomic emission spectroscopy (ICP-AES). Digested solution was filtered and used for ICP AES analysis after dilution. While carrying out ICP AES analysis, readings in triplicate were ensured for error minimization. The concentration as achieved by ICP AES analysis was multiplied with dilution factor to find out concentration in digested volume. Concentration, hence achieved, was multiplied with the initial volume to measure the mass of individual metal present in the filtrate giving milligram of metal per unit gram of pre-processed PCB material.

3.2.4 Silver and Copper Extraction

4 Molar Nitric Acid was used to leach out silver and copper from pre-processed PCB material. A liquid to solid ratio of 10:1 was kept to ensure sufficient acid availability and surface exposure to granulated material. For homogenous mixing during reaction, a Well Stirred Batch Reactor (WSBR) was designed using 2 litre volume flask; a thermometer was installed as temperature probe. Hot plate magnetic stirrer was used to provide continuous mixing and heat. A temperature in the range of 65 to 68°C was maintained throughout the

reaction period. 100 gram sample material was used for leaching experiment to extract the silver and copper. 1 litre of 4 M Nitric Acid was prepared. Sample was first placed into reactor. 4 M Nitric Acid solution was poured slowly to ensure that bubble formation and fumes were not forcing the solution to come out of reactor. Magnetic stirrer was kept open to maintain homogeneous mixing. Initially due to exothermic reaction, the reactor temperature reaches 70°C and as reaction proceeds it was maintained by external heat. Initially to standardise the optimum reaction time for maximum leaching of desired metal, 10 ml of reaction mixture was taken out after 15 minutes of reaction initiation. After every 30 minutes, 10 ml reaction mixture was taken out of reactor and filtered instantly. As it was a well-mixed reactor, the liquid to solid ratio will remain constant in reactor. Filtered solution was analysed for desired metals using ICP AES to find out the increase in concentration with time. During the progression of reaction, there will be loss in volume due to evaporation. For eliminating the increase in concentration due to evaporation error, a blank run of well-mixed reactor using 4 molar Nitric Acid was carried out and after initial 15 minutes and every subsequent 30 minutes, loss in volume was measured. The error correction was taken into account for finding out optimum reaction period for maximum silver-copper leaching. After completion of reaction, solution was filtered to remove remaining solid particles. The solids so removed were kept at 100°C for removing moisture while the filtered (pregnant) solution was analysed using ICP AES for metal concentration. Concentration so achieved was multiplied by the total volume to calculate mass of individual metal present in solution, extracted from 100 gram of initial pre-processed PCB material. Solid material so received will be utilised for extracting the Gold in further extraction steps.

3.2.4.1 Recovery of Silver

Pregnant solution containing copper and silver was filtered to remove any remaining residue. 50wt% sodium chloride (NaCl) was added to pregnant solution. After mixing, Silver should form the precipitate of silver chloride (AgCl). To remove the impurities of Lead Chloride and Mercury Chloride, precipitate should be washed with 0.5 M Nitric Acid. 10% Potassium Hydroxide solution will be added and mixed for 30 minutes at 25°C to form Silver oxide. Silver oxide produced will be added to 30% Hydrogen Peroxide solution to form metallic Silver powder. Metallic silver powder will be separated by filtration. An alternate approach of zinc cementation was also used for recovery of Silver. Zinc powder with mass ratio of 30:1

was added to precipitate out silver. Recovery efficiency was calculated based on decrease in the concentration of Silver before and after recovery protocol.

3.2.4.2 Recovery of Nitric Acid

Pregnant solution contains excess Nitric Acid ready to be recovered using solvent extraction process with Tri Butyl Phosphate (TBP) dissolved in Kerosene. Mechanical shaking of 250 ml of 50% Tri Butyl Phosphate and 250 ml of pregnant solution was done to remove excess nitric acid which can be further used in extraction. Phase separation was done using separating funnel. Aqueous phase now mainly contains Copper which will be used as electrolyte in electro-deposition experiments.

3.2.4.3 Recovery of Copper

Copper and Stainless Steel as well as lead plates with size 7 cm x 8 cm were used as electrodes in an assembly size of 8 cm x 8 cm x 15 cm. Current was supplied using a DC power source for 24 hour at room temperature. Current density of 45 mA/cm^2 was maintained. Copper was deposited on cathode creating a decrease in concentration of copper in solution. Efficiency of copper recovery was calculated based on decrease in concentration of copper after electro-deposition while considering the decrease in volume of initial feed.

3.2.5 Complete Digestion of Remaining Solids

Solids left after nitric acid leaching were first dried at 100° C temperature. Complete digestion was performed to calculate remaining individual metals in sample. A different acid combination was used to digest the remaining solids as the earlier combination was not successful. Hydrochloric Acid was included in combination to make the conditions more oxidative. Various combination of Nitric Acid, Hydrochloric Acid, Hydrogen peroxide and Hydrofluoric Acid with final 14 ml acid volume were tested for digesting 1 gram sample. 6 ml Nitric Acid, 4 ml Hydrochloric Acid, 2 ml Hydrogen Peroxide and 2 ml Hydrofluoric Acid or a ratio of 3:2:1:1 was successful.

3.2.6 Extraction of Gold

Thiosulfate leaching has been used as a preferred leaching agent for gold due to its environmental friendly behaviour. Leaching of gold in ammonium thiosulfate solution is an electrochemical reaction with catalytic effects of cupric ions. 1 Molar Ammonium Thiosulfate, 0.1 Molar Copper Sulphate and 5 M Ammonium Hydroxide were used to extract the gold from solids as received after copper and silver extraction. A solid to liquid ration of 1:10 was maintained to ensure sufficient exposure and optimum condition for leaching. Temperature of 40°C was constantly maintained throughout the reaction. Homogeneous mixing was ensured through magnetic stirring of 500 rpm. After 30 hour reaction period, solution was filtered to separate solids and pregnant solution. The filtrate was analysed using ICP AES. Solid residue left were taken forward for complete digestion to calculate remaining gold in solid. Based on initial and remain gold concentration, efficiency of extraction was calculated.

3.2.6.1 Recovery of Gold

Cementation was used for gold recovery from pregnant solution. Zinc cementation is the process of cathodic deposition of gold and anodic corrosion of zinc at the surface of zinc particles. Zinc metal powder (45 micron) was added to the solution in optimum mass ratio 30:1. Various mass ratio of Zinc to gold were tested to find optimum zinc requirement for gold cementation. With adequate mixing, gold will precipitate and settle at the surface. A constant mixing at 400 rpm was maintained throughout the cementation process.

Chapter 4
Results and Discussion

4.1 Sample Preparation

While many research studies shows the use of Ball Mill and jaw crusher for the comminution of Printed Circuit Boards, it was observed in current study that both Ball Mill and Jaw Crusher processing were inefficient in achieving homogeneous comminution. This can be explained with the ductile nature of Printed Circuit Boards while the mentioned instruments have been specifically designed for brittle materials like rock etc. After PCB shredding, shredded material was fed to Ball Mill for 4 hour. Output material lacked surface mountings of PCB and the processing lead to size reduction of mounted material along with some base platform. After this operation, the sample was sieved through 1 mm sieve, particles so collected were below 1 mm size and named Sample A. Sample above 1 mm particle size was fed to grinding cutter. The output was fractioned based on density into three portions: one metal rich other resin as well as plastic rich sample. These samples were named Sample B, Sample C and Sample D. So PCB material was divided into four fractions to recover metals from PCB. Particle size range for Sample A and B was ≤ 1 mm while sample C and D ranged between 1 mm to 2.5 mm.

4.2 Optimization of Reactants in Microwave Digester

To standardize the digestion protocol, the combination of acids were optimised for achieving complete digestion. The samples were digested using different volumes of Nitric Acid, Hydrogen Peroxide and Hydrofluoric Acid in a Microwave Digester. The success of digestion was determined based on a clear solution without any black shed or particle as output. Table 4.1 summaries various process tested and their qualitative outcome.

Table 4.1 Combinations of Acid for Complete Digestion of Sample

Test No.	Nitric Acid (ml)	Hydrofluoric Acid (ml)	Hydrogen Peroxide (ml)	PCB Sample (Gram)	Remarks
1.	10	3	1	1	Unsuccessful
2.	9	3	2	1	Unsuccessful
3.	8	3	3	1	Unsuccessful
4.	8	2	4	1	Unsuccessful
5.	8	4	2	1	**Successful**

4.3 Quantification of Initial Metal Concentration

Digested sample were analysed for metal content mainly Copper, Silver, Gold, Palladium and Platinum using ICP AES. ICP AES provides concentrations in parts per million or milligram per litre. The concentration received in ppm was multiplied with dilution factor and the initial volume to get the mass of individual metal per gram of sample. While copper, silver and gold were detected in ICP Analysis, Platinum and Palladium remained below detectable limits (0.001 ppm) of ICP AES. Tables 4.2, 4.3, 4.4 and 4.4 show the average metal concentration along with standard deviation for Samples A, B, C and D. Metal distribution in homogeneously powdered printed circuit boards cannot be called homogeneous because of specific metal application in specified parts of PCB making it difficult to compute an exact estimation of individual metal concentration. For calculating representative individual metal concentrations, experiments were performed in triplicate while ensuring triplicate ICP readings of each sample to maintain nine fold error minimization.

Table 4.2 Initial Metal Content in Sample A

Metal	Concentration (mg/gram)	Standard Deviation
Copper	178.003	11.065
Silver	0.1522	0.079
Gold	0.193	0.041

Table 4.3 Initial Metal Concentration in Sample B

Metal	Concentration (mg/gram)	Standard Deviation
Copper	284.876	30.843
Silver	0.1490	0.011
Gold	ND#	ND

Table 4.4 Initial Metal Concentration in Sample C

Metal	Concentration (mg/gram)	Standard Deviation
Copper	152.622	12.454
Silver	0.0279	0.005
Gold	ND	ND

Table 4.5 Initial Metal Concentration in Sample D

Metal	Concentration (mg/gram)	Standard Deviation
Copper	206.359	17.897
Silver	0.0509	0.0083
Gold	ND	ND

#ND: Below Detectable Limit (<0.001 ppm)

Below detectable limit concentration of gold in sample B, C and D was expected as most of the mountings were crushed in Ball Mill (Sample A) while base platform of printed circuit boards was processed by grinding. As most of the gold applications in PCBs are for connections with high conductivity, it is likely that it would have gone away in Ball Mill processed sample A of < 1 mm particle size range.

4.4 Hydrometallurgical Processing

4.4.1 Extraction of Copper and Silver

Pre-processed samples were subjected to Nitric Acid leaching to extract Silver and Copper as per the details given in section 3.2.4. Figure 4.1 shows the copper extraction profile of Sample A in triplicate run R1, R2 and R3 while Figure 4.1 R shows the representative copper extraction profile along with standard deviation. First two reading were taken after 15 minutes interval while subsequent readings after 30 minutes each, till a total time of 240 minutes. Figure 4.2 provides extraction profile for three runs for silver in Sample A Same procedure was followed to analyse the extraction rate profiles of Samples B, C and D.

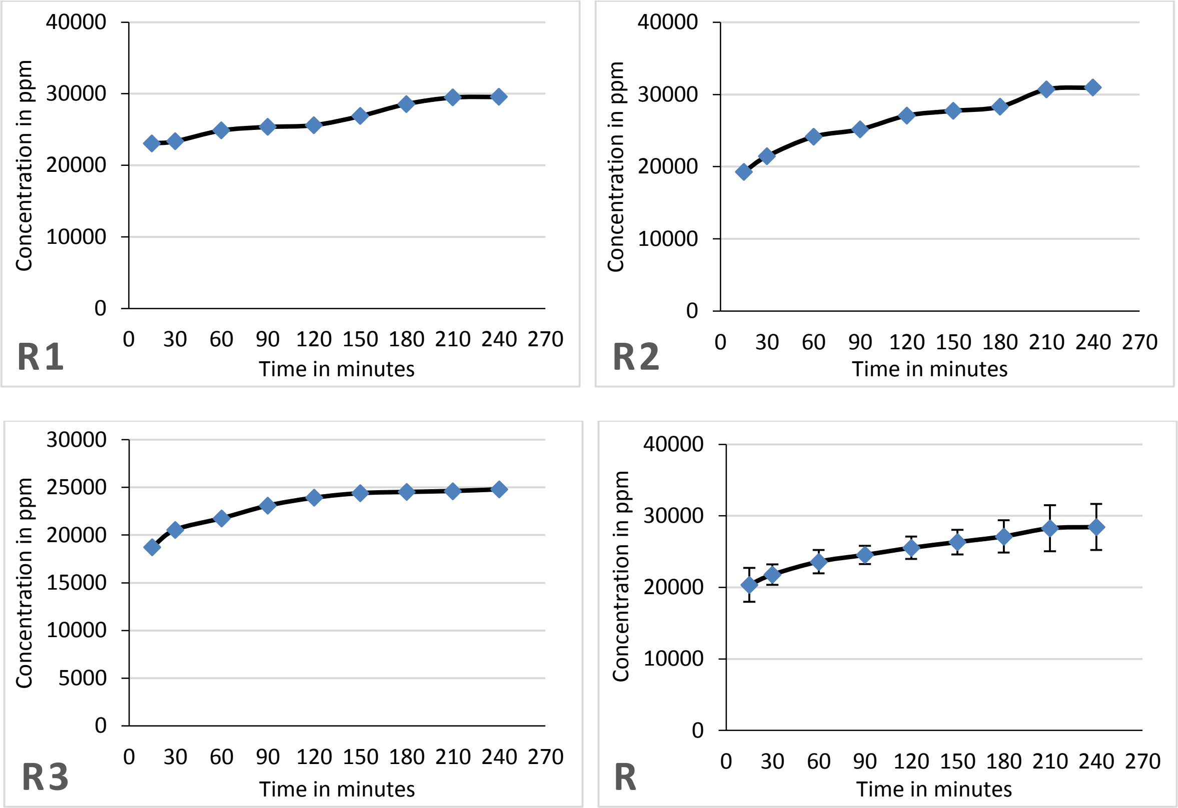

Figure 4.1 Copper Extraction Profile of Sample A with Time

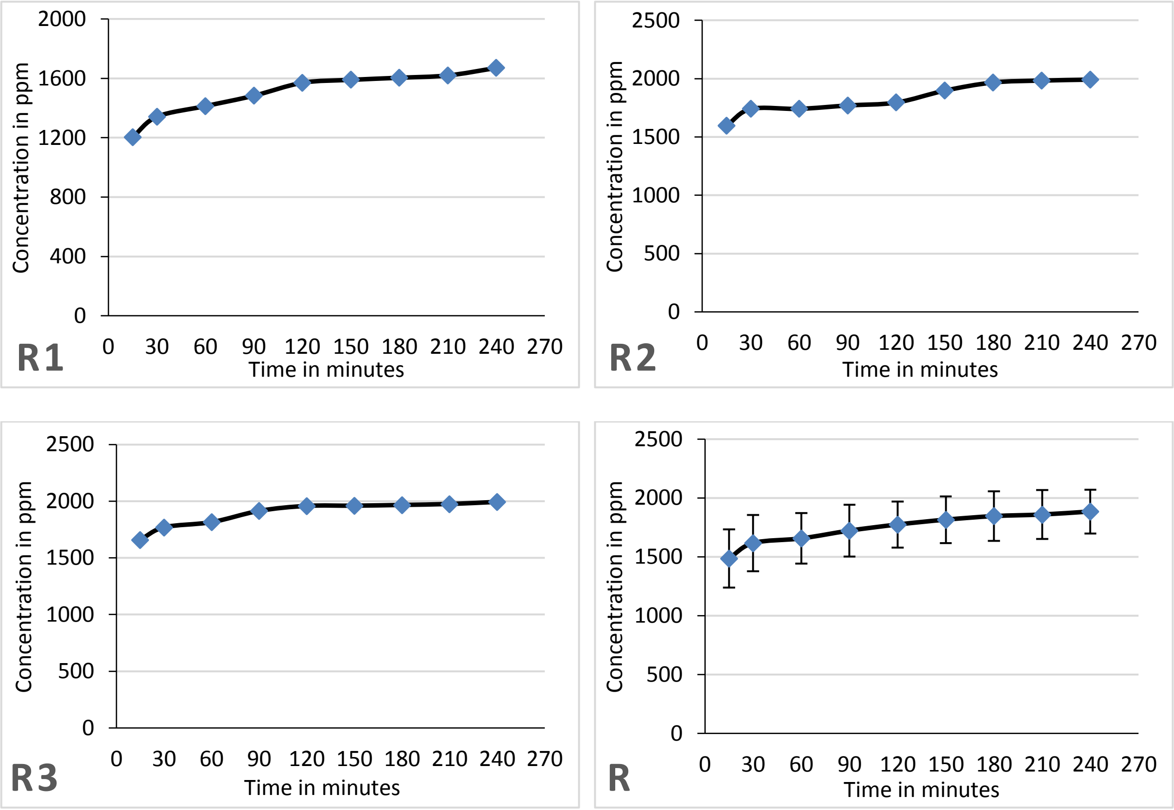

Figure 4.2 Silver Extraction Profile of Sample A with Time

Metal extraction experiments were also analysed to find optimum time for maximum extraction. It can be observed from Figure 4.1 that after 210 minutes, the concentration reaches a near plateau which can be described as optimum time for this sample. Figure 4.2 R1, R2 and R3 illustrate increase in silver concentration for individual runs while Figure 4.2 R combines the average concentrations for all three runs at each interval point along with standard deviation to provide characteristic extraction profile for silver with highest average concentration being 1885.1 ppm with a standard deviation of 186.3.

Figure 4.3 highlights the copper extraction profile for Sample B. Triplicate can be observed through Figure 4.3 A, B and C having maximum concentration of 32800.5 ppm, 33106.5 ppm and 35756.5 ppm respectively at 240 minutes. Representative extraction profile shows a maximum average concentration of 33887.8 with standard deviation 1625.5.

Silver extraction profile for Sample B has been shown in Figure 4.4 with triplicate runs R1, R2 and R3. Figure 4.4 R shows the representative profile with highest average silver concentration of 1708.6 ppm with standard deviation of 135.6 ppm at 240 minutes a little higher compared to 210 minutes.

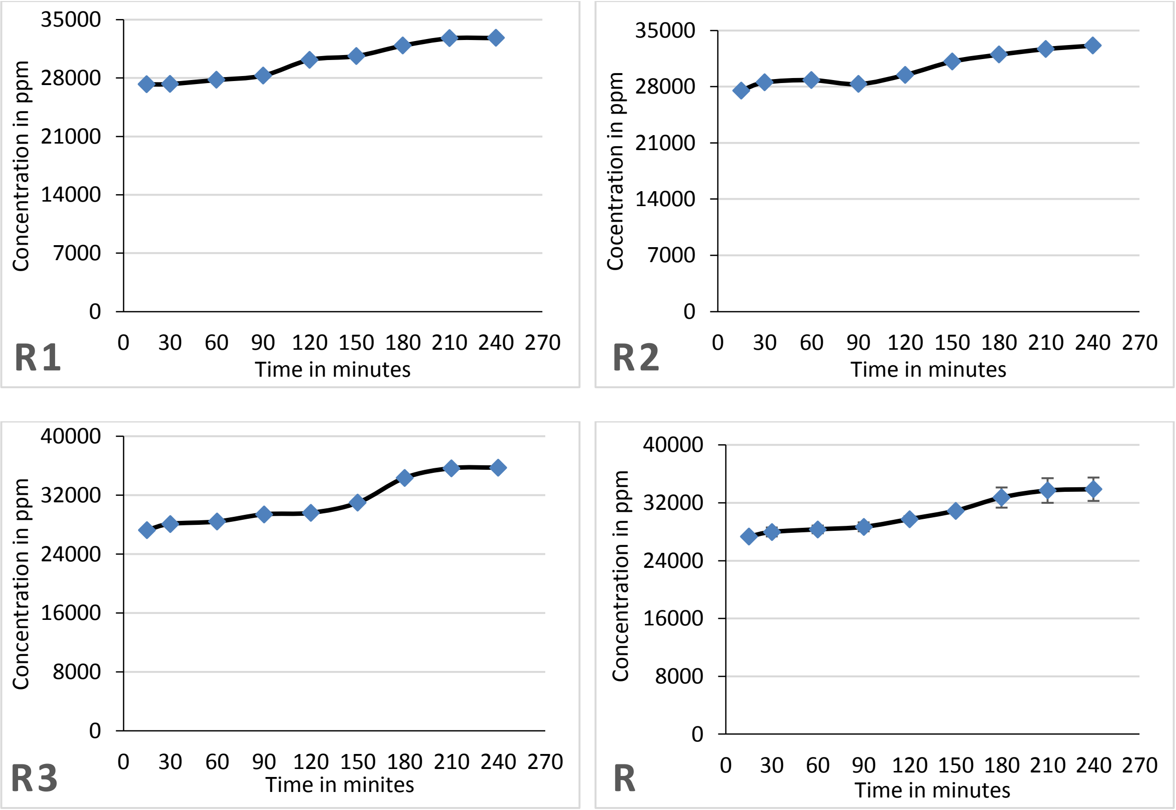

Figure 4.3 Copper Extraction Profile of Sample B with Time

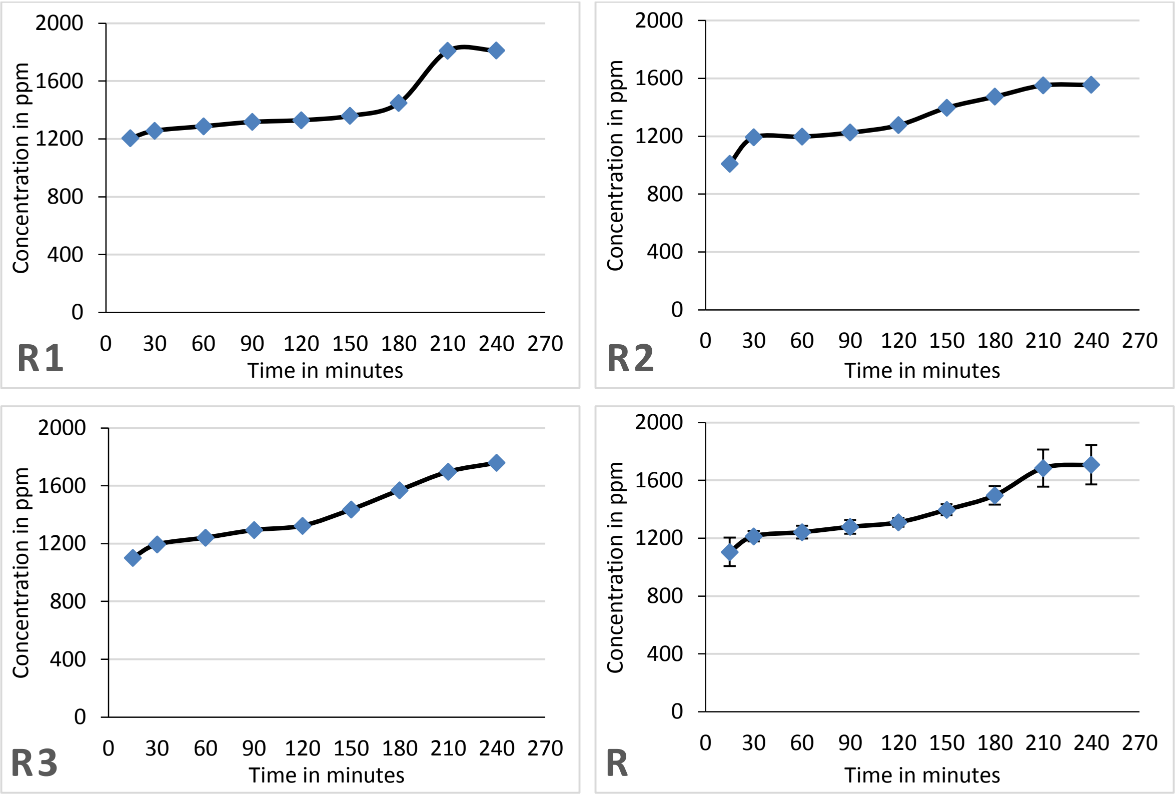

Figure 4.4 Silver Extraction Profile of Sample B with Time

Figure 4.5 and Figure 4.6 depicts change in copper extraction rate profiles for Sample C and D. Higher variation in slope of these figures can be explained through particle size properties. While Sample A and B, both were in size range of ≤ 1 mm, the surface to volume ratio was high, leading to maximum possible exposure of acid to react with metals and extract them out. As discusses earlier, particle size for Sample C and D was in the range of 1 mm to 2.5 mm. In case of Sample C and D, though the comparative concentration is higher, metal remains in deeper portions of big particles leading to slow initial extraction, increasing with time as the outer surface gets more and more exposed to acid. Figure 4.5 R1, R2 and R3 describe triplicate runs for Sample C with highest concentration of 64185 ppm, 68097.5 ppm and 61050 ppm while maximum average concentration for representative profile was 64444.1 ppm with standard deviation of 3530.8 as shown in Figure 4.5 R.

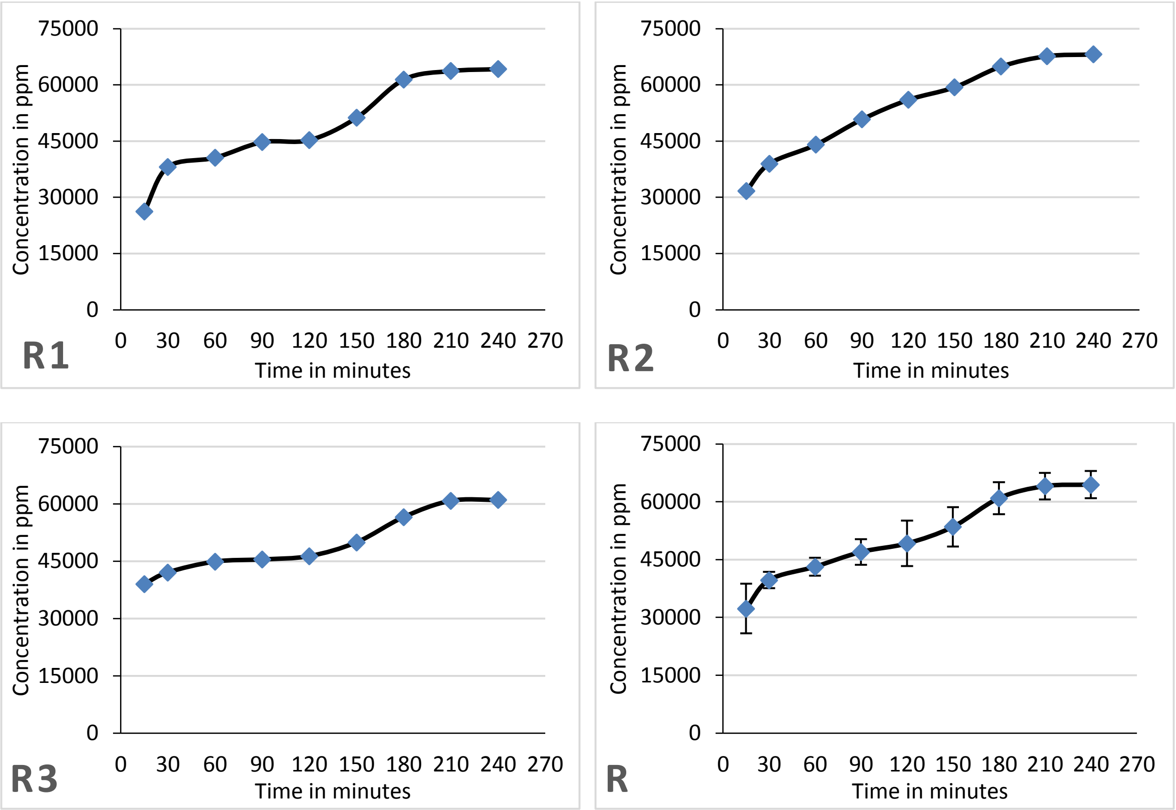

Figure 4.5 Copper Extraction Profile of Sample C with Time

Figure 4.6 R1 provides leaching rate profile of first run of the sample for copper extraction. The highest concentration of 51501.5 ppm was achieved for run R1 while for run R2 and R3, maximum concentrations were 50051.5 ppm and 50651 ppm. Average maximum concentration for representative profile was 50734.6 ppm with standard deviation of 728.6 as shown in Figure 4.6 R.

It can be observed from the silver extraction profiles of Sample C and D in Figure 4.7 R1 and 4.7 R2 that the extracted silver is very less. Due to low concentrations and high standard deviation, individual runs are not presented. Maximum average silver concentration extracted from Sample C was 20.4 ppm with standard deviation of 7.3 while for Sample D it was 8.6 ppm with a standard deviation of 4.7. Low concentrations and high standard deviations may have its justification on low initial concentrations of silver in sample C and D being rich in resin and plastic.

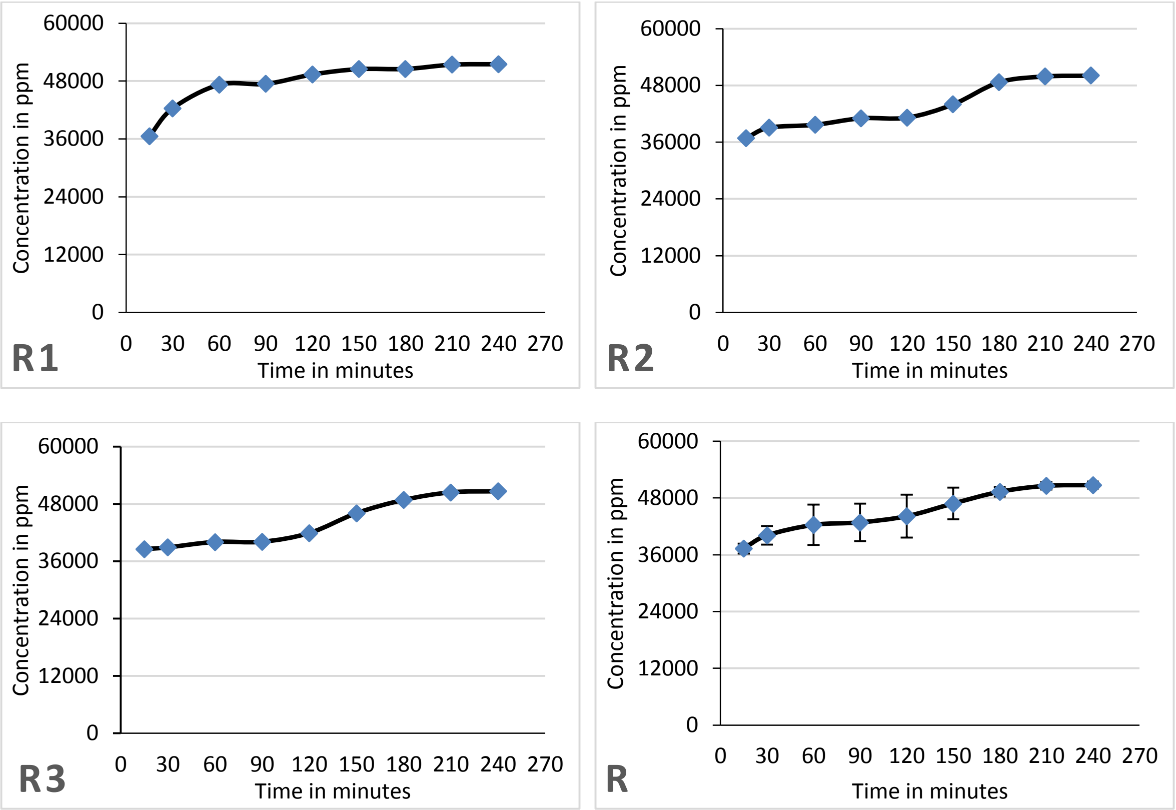

Figure 4.6 Copper Extraction Profile of Sample D with Time

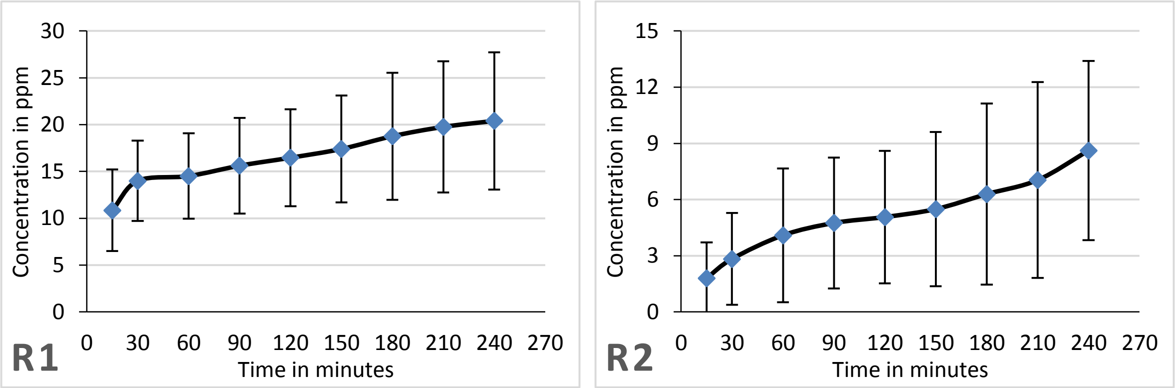

Figure 4.7 Rate of Silver Extraction with Time for Sample C (R1) and Sample D (R2)

Table 4.6 and 4.7 shows the extraction efficiencies of nitric acid leaching for copper and silver. Efficiency has been calculated after the complete digestion of remaining solids. It can be observed that the copper extraction efficiency ranges from 91.9% to 96.8% while for silver, the extraction efficiency of 97.4% to 99.9% has been achieved.

Table 4.6 Copper Extraction Efficiency of Nitric Acid Leaching

	Initial Copper Concentration (mg/gram)	**Standard Deviation**	**Post-extraction Copper Concentration (mg/gram)**	**Standard Deviation**	**Extraction Efficiency (%)**
Sample A	178.004	11.066	5.697	0.223	96.8
Sample B	284.876	30.843	12.543	3.276	95.6
Sample C	152.622	12.454	8.054	3.031	94.7
Sample D	206.359	17.897	16.726	2.322	91.9

Table 4.7 Silver Extraction Efficiency of Nitric Acid Leaching

	Initial Silver Concentration (mg/gram)	**Standard Deviation**	**Post-extraction Silver Concentration (mg/gram)**	**Standard Deviation**	**Extraction Efficiency (%)**
Sample A	0.1522	0.0793	0.000123	0.000018	99.9
Sample B	0.1490	0.0118	0.001118	0.000081	99.2
Sample C	0.0279	0.0056	0.000721	0.000255	97.4
Sample D	0.0509	0.0084	0.000827	0.000348	98.3

4.4.1.1 Recovery of Silver

Pregnant leach solutions achieved after extraction process were further subjected to metal recovery. As mentioned earlier in Chapter 3, first silver needs to be recovered from solution as the electro-deposition of copper can recover silver as impurity along with copper deposition. For silver deposition 50wt% Sodium Chloride solution was added to the pregnanat solution. As per literature, silver should form the white precipitate while it was not observed in current study even after excessive addition of NaCl. An alternate approach of zinc cementation was used to recover silver from pregnant solution. First, 30:1 mass ratio of zinc powder was added but there was no sign of precipitation. Additional zinc powder was added and sample was collected for ICP analysis. Table 4.8 shows decrease in silver concentration with addition of zinc powder. With excessive zinc addition, the decrease in concentration was only 4.3%. This can be concluded that it was an unsuccessful approach in this case.

Table 4.8 Silver Removal by Zinc Cementation

	Silver Concentration (ppm)	**Zinc Powder (gram)**
Sample	63.752	0.5
	63.636	1
	61.532	1.5
	61.005	2
Percent Decrease (%)	4.3	-

4.4.1.2 Recovery of Copper

For copper recovery, electrowinning assembly as shown in Figure 4.8 was used. With a current density of 45 mA/cm^2 and deposition time of 24 hours separate runs were performed. Copper was kept with negative charge as cathode while stainless steel and lead were used as anodic electrode. Literature suggest that nitric acid interferes with the copper deposition. To verify, deposition experiments were performed with pregnant solution without nitric acid removal and later with nitric acid removal by solvent extraction process. For recovery experiments, only triplicates of Sample A and Sample B were used. Table 4.9 shows the recovery efficiency for three samples namely B1, B2 and B3. For sample B1, lead was used as anode while copper plate was used as cathode. For sample B2 and B3, Stainless Steel was used as anode while Copper electrode was used as cathode. From Table 4.9, it can be observed that highest recovery efficiency has been achieved by lead and copper electrode combination which is 80.2% while for stainless steel and copper combination the efficiency were 74.5% and 74.9% respectively.

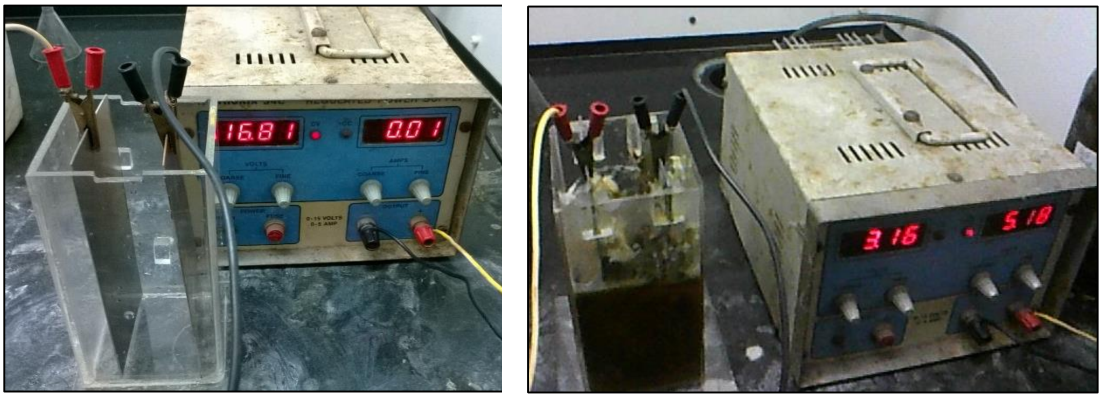

Figure 4.8 Copper Electrowinning Assembly

For better copper recovery, nitric acid was extracted from pregnant solution using solvent extraction process. An average of 40 ml nitric acid was extracted with two stage solvent extraction using Tributyl Phosphate in kerosene. Table 4.10 shows the recovery efficiency of copper, 89.6%, from aqueous phase of solvent extraction using 45 mA/cm^2 current density at stainless steel anode and copper cathode for 24 hours.

Table 4.9 Copper Recovery Efficiency of Electro-deposition from Nitric Acid Rich Solution

	Post-recovery Copper Concentration (ppm)	**Mass Remaining (mg)**	**Initial Copper Concentration (ppm)**	**Initial Mass (mg)**	**Efficiency Based on Mass Recovery (%)**
Sample B1#	16203.2	3240.64	32800	16400	80.24
Sample B2	21508.4	4301.68	33200	16932	74.59
Sample B3	19081.72	3816.34	35800	15215	74.91

#Lead electrode as Anode

Table 4.10 Copper Recovery Efficiency of Electro-deposition after Nitric Acid Removal

Sample	**Post-recovery Copper Concentration (ppm)**	**Mass Remaining (mg)**	**Initial Copper Concentration (ppm)**	**Initial Mass (mg)**	**Efficiency Based on Mass Recovery (%)**
Sample A	15315.7	1531.57	32836.66	14776.497	89.6

4.4.2 Extraction of Gold

Table 4.11 shows the initial concentration of gold in solid residues left after nitric acid leaching. Based on concentration, only residues left out of Sample A triplicate runs were selected for gold extraction process. Three parts of original Sample A found to have gold concentration from 0.19745 mg/gram to 0.39886 mg/gram. Supporting initial gold concentration analysis, Sample B, C and D possessed very less gold from below detectable limit of ICP to 0.05137 mg/gram with even higher standard deviation of 0.07728. Based on this, only three subsamples of Sample A, were taken forward for further experiments.

Table 4.11 Initial Gold Concentration in Solid Residue

Sample Name	**Gold Concentration (mg/gram)**	**Standard Deviation**	**Selection for Gold Leaching**
Sample A Part-1	0.39886	0.14892	**Selected**
Sample A Part-2	0.25861	0.07408	**Selected**
Sample A Part-3	0.19745	0.23361	**Selected**
Sample B Part-1	0.02134	0.01973	Not Selected

Sample B Part-2	BDL#	-	Not Selected
Sample B Part-3	BDL	-	Not Selected
Sample C Part-1	0.01397	0.02419	Not Selected
Sample C Part-2	0.05137	0.07728	Not Selected
Sample C Part-3	0.04642	0.08040	Not Selected
Sample D Part-1	BDL	-	Not Selected
Sample D Part-2	BDL	-	Not Selected
Sample D Part-3	BDL	-	Not Selected

#BDL: Below Detectable Limit

Figure 4.9 shows gold extraction profile from subsamples of Sample A. Thiosulfate leaching was used to extract gold from printed circuit board samples. 30 hours treatment was given to sample in a liquid to solid ratio of 10:1. Figure 4.9 R1 shows the increase in concentration of gold with time in hours. Three samples, each after 10 hour's interval, were collected for gold concentration analysis by ICP. The concentration values for all three run can be seen in Figure 4.9 R1, R2 and R3.

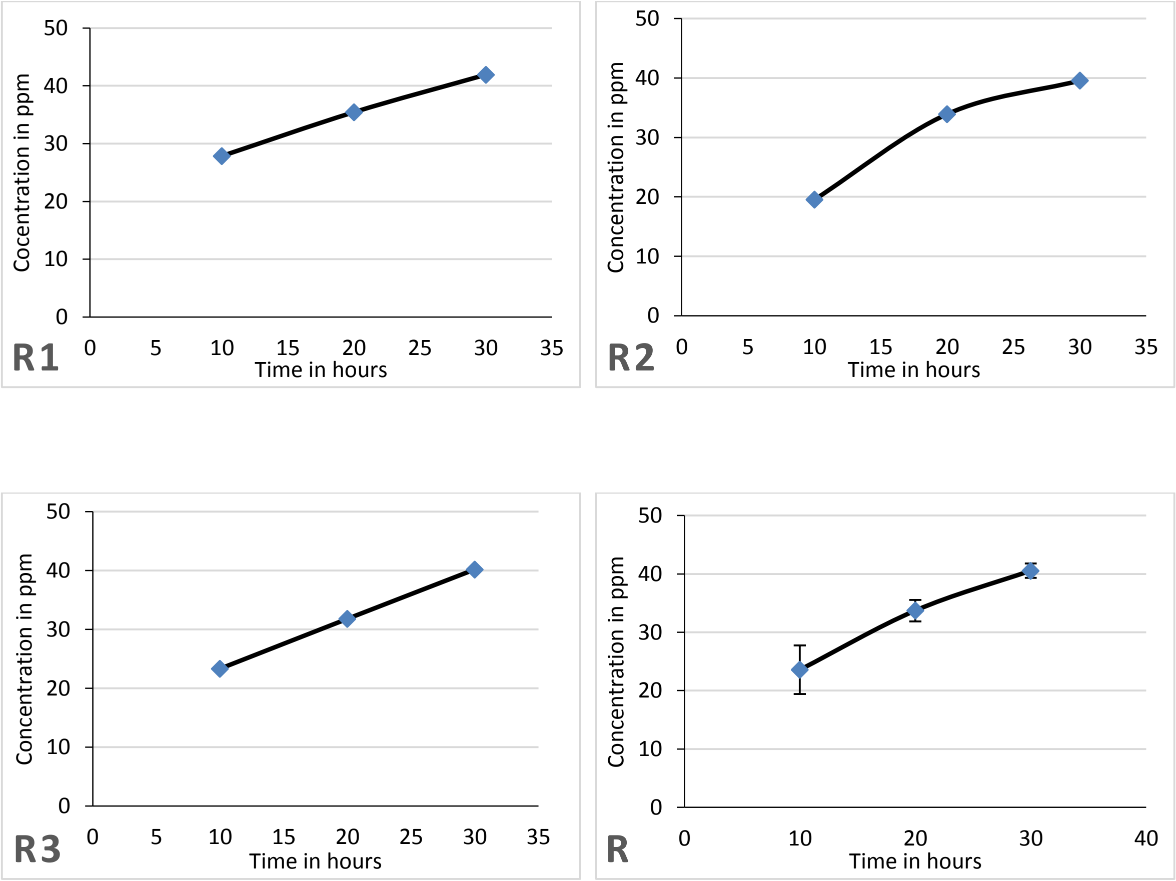

Figure 4.9 Increase in Concentration with Time for Gold Extraction

Figure 4.9 R1 shows a maximum concentration of 41.9 ppm at 30 hours while in run R2 and R3, maximum gold concentration achieved was 39.5 ppm and 40.1 ppm. For three runs, a representative leaching profile was prepared with average at sampling time interval which has been shown in figure 4.8 R. The maximum average gold concentration after extraction was 40.5 ppm with standard deviation of 1.2. Table 4.12 shows the extraction efficiency of thiosulfate leaching for gold in this study. Efficiency was calculated based on decrease in total mass of gold in whole sample. Efficiency ranged from 55.0 to 74.3%.

Table 4.12 Efficiency of Gold Extraction Calculated Based on Total Mass

	Gold Concentration Remaining after Extraction (mg/gram)	**Mass of Gold Remaining in Total Sample (mg)**	**Initial Mass of Gold in Total Sample (mg)**	**Extraction Efficiency (%)**
Sample 1	0.0706	3.75	14.6398	74.3
Sample 2	0.1254	6.58	14.6398	55.0
Sample 3	0.0807	4.96	14.6398	66.1

4.4.2.1 Recovery of Gold

Gold so extracted was subjected to zinc cementation for recovery. Table 4.13 shows the efficiency of zinc cementation for gold recovery. Cementation for each sample was performed in triplicate. Based on decrease in concentration after cementation efficiency of recovery was calculation

Table 4.13 Gold Recovery Efficiency of Zinc Cementation

Sample	**Initial Gold Concentration (ppm)**	**Post-Cementation Concentration (ppm)**	**Average Post-Cementation Concentration (ppm)**	**Efficiency of Recovery (%)**
Sample 1.1	41.9	5.5	5.8	85.9
Sample 1.2		6.2		
Sample 1.3		5.8		
Sample 2.1	39.5	7.2	7.5	80.9
Sample 2.2		7.3		
Sample 2.3		8.0		

Chapter 5

Conclusions and Future Scope of Work

Continuous increase in electronic waste generation along with decreasing life time of electrical and electronic equipment enforces the need of environmentally sound management of waste electrical and electronic equipment. Recycling of electronic waste and recovery of metals provides economically beneficial solution for e-waste problem. There is a huge gap between the amount of e waste generated and fraction recycled. Technologies deployed for resources recovery from electronic waste are very harsh and produce severe environmental health and safety issues. The efficiency of metal recovery from electronic waste is also lower because of manual handling and absence of proper protocols. Physical method of metal recovery from electronic waste provides various steps to separate ferrous and non-ferrous metals, plastic and other resources. Metallurgical methods for metal recovery are most commonly followed in the formal and well as informal sector of electronic waste recycling. While pyro-metallurgy has been the key field for large scale recyclers, it may not be possible for small scale recyclers to accept it as their technology. Biometallurgy is in very naïve form with major research and applications in mineral processing. With an ease of operation, control and feed requirement, Hydrometallurgical methods remain important hope for small scale recyclers. In pursuit of finding an environmentally sound technology for metal recovery from electronic waste, current study deals with recovery of copper, silver and gold from printed circuit boards of personal computers.

Major observations can be summarized as:

1. Ball mill and jaw crusher are not effective means for homogeneous size reduction of printed circuit boards which is primarily made up of ductile material.
2. A 4:2:1 ratio of Nitric Acid, Hydroflouric Acid and Hydrogen Peroxide best suits the sample in current study for complete digestion using microwave digester.
3. For selective leaching of copper and silver, 4 Molar Nitric Acid acts as an efficient leaching agent.
4. Extraction efficiency in the range of 91.9% to 96.8% were achieved for copper.
5. Extraction efficiency in the range of 97.4% to 99.9% were achieved for silver.

6. For recovery of silver from nitric acid rich solution, sodium chloride precipitation and zinc precipitation are not recommended methods. There is a need of further research to identify suitable method.
7. Recovery of nitric acid increased the copper recovery from 74.5% to 89.6%. While use of lead electrode gives higher efficiency of recovery, 80.2%, it has higher electrode dissolution rate which will add lead into remaining waste after metal extraction.
8. Lead being a serious concern as a pollutant and costly compared to stainless steel, it may not be advisable to use it in metal recovery.
9. Solvent Extraction process for nitric acid not only increases the copper recovery efficiency but also provides nitric acid for use in next cycle of metal extraction along with reducing the hazardous nature of waste remaining after the metal extraction. So, this approach have three fold benefits while minimizing overall environmental threats of hydrometallurgical approaches.
10. Current study is one of the very early study to carry out gold extraction from electronic waste using thiosulphate. An extraction efficiency of 55% to 74.3% has been achieved.
11. Zinc cementation has been successfully applied for recovery of gold from pregnant solution. Maximum cementation efficiency of 85.9% has been achieved.
12. Process of thiosulfate leaching and cementation were adopted from mineral processing practices which confirms the validity of mineral processing techniques in metal recovery from electronic waste.

5.1 Future Scope of Work

On the basis of results achieved in this study, it is possible to convert this methodology into a working technology for small scale recyclers. Such a conversion will require additional research to ensure minimum scale up problems along with further validation. This work was carried out on terms of worst case scenario keeping in mind informal recycler. No metal recovery was done with mechanical methods to keep the extraction conditions more complex. Metals like iron and aluminium were not separated before size reduction to maintain worst case scenario treatment. Specific work could be done after mechanical processing of waste to recover easily extractable metals like iron and aluminium using magnetic separation. Nonmagnetic metallic fraction treatment with the methodology developed could lead to better recovery efficiency. Specific problems of this work needs further research. There is a need of silver recovery technique from nitric acid rich solutions. There is need for extensive research on application of thiosulfate for gold leaching from variety of electronic waste to create more optimum dose information which can be scaled up as a small-medium scale industry.

Presentations and Publications

Arora, M. and Dikshit, A.K. (2014) Combined Hydrometallurgical Approach towards Metal Recovery from Printed Circuit Boards: An Organised Technique for Unorganised recyclers in Electronic Waste Management. *Proceedings of E-Waste Academy Scientist Edition, Basel Convention, United Nations University and Shanghai Second Polytechnic University, Shanghai.*

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URL-03: <http://www.greenpeace.org/international/en/campaigns/toxics/electronics/the-e waste-problem/where-does-e-waste-end-up/> (accessed on 29.11.2014)