

**RECOVERY OF METALS FROM WASTE PRINTED
CIRCUIT BOARDS USING LOW-TEMPERATURE
AMMONIUM CHLORIDE ROASTING**

by

**RAMDAYAL PANDA
DEPARTMENT OF CHEMICAL ENGINEERING**

**Submitted
In fulfilment of the requirement of the degree of
DOCTOR OF PHILOSOPHY
TO THE**



**INDIAN INSTITUTE OF TECHNOLOGY DELHI
DECEMBER 2022**

Certificate

This is to certify that the thesis entitled “**Recovery of Metals from Waste Printed Circuit Boards Using Low-Temperature Ammonium Chloride Roasting**” submitted by **Mr. Ramdayal Panda** to the Indian Institute of Technology Delhi, for the award of the degree of **Doctor of Philosophy**, is a record of the original bonafide research work carried out by him. He has worked under my supervision and has fulfilled the requirements, which to my knowledge, has reached the requisite standard for the submission of this thesis. The results contained in this thesis have not been submitted in part or full to any University or Institute for the award of any degree or diploma.



Dr. Kamal K. Pant

Professor

Department of Chemical Engineering
Indian Institute of Technology Delhi



Dr. Satya N. Naik

Professor

Center for Rural Development and Technology
Indian Institute of Technology Delhi



Dr. Thallada Bhaskar

Senior Principal Scientist

Material Resource Efficiency Division
CSIR- Indian Institute of Petroleum Dehradun

Acknowledgments

It's truly been an incredible journey full of adventure, which would not have been possible without the support and encouragement of many people. First and foremost, I would like to express my gratitude and special appreciation to my supervisor Prof. Kamal K. Pant, who provided me guidance, valuable suggestions, and motivation throughout my Ph.D. journey. Also, I would like to express my special appreciation and warm regards to my co-supervisors Prof. Satya N. Naik (Centre for Rural Development and Technology, IIT Delhi) and Prof. Thallada Bhaskar (Material Resource Efficiency Division, CSIR- IIP Dehradun) for their valuable suggestions, motivation, and keen interest throughout my research work.

I am thankful to Prof. A. K. Saroha, Head of the Chemical Engineering Department, for providing the necessary infrastructure and laboratory facilities to carry out research work. Also, I would like to thank all my SRC members: Prof. S. Upadhyayula, Prof. D. Bhatia, and Prof. A. K. Nema for their encouragement, insightful comments, and suggestions.

I express my special thanks to my senior lab members Dr. Sonal Asthana, Dr. Ejaz Ahmad, Dr. Arindam Modak, Dr. Shireen Qureshi, Dr. Tapas Dora, Dr. Ashish Bohre, Dr. Kunwar Pal Dr. Rohit Kumar, Dr. Sonit Balyan, Dr. Ashish Pandey, Dr. Kaushal Parmar, Dr. Sourabh Mishra, Dr. Prashant Ram Jadhao, Mr. Akshay Mankar, Mrs. Uma Dwivedi for their support and encouragement during my research work. Also, Ms. Komal Tripathi, Mr. Rajan Singh, Mr. Sagar Dhanushkar, Ms. Snigdha Mishra, Ms. Shally Gupta, Ms. Amrita Preetam, Ms. Shreya Singh, and all other research scholars from Catalytic Reaction Engineering Laboratory for their support and motivation. I would like to extend my thanks to Mr. Vishesh Kumar, Mr. Krishna Kumar, Mr. Suchit Kumar, Mr. Ashish Pal, and Mr. Vijay Pal for their constant help in the smooth conduct of my research work.

I would like to dedicate this thesis to my father, Mr. Surendra Mohan Panda, my mother, Mrs. Minati Panda, and my sister, Ms. Tosatmika Panda for their love, blessing, and enormous trust. I feel blessed to have them on every path and journey of my life.

I also want to express my sincere thanks to all those who had directly or indirectly helped me in the completion of my research.

Finally, I thank the almighty God for his blessing and for giving me strength throughout the journey of life.



Ramdayal Panda

Abstract

Circular economy is not a choice but a necessity for every sector with growing demand and limited resources. Electronic waste (e-waste) is currently one of the most hazardous solid wastes posing a serious threat to the environment, primarily due to its high generation rate, further complemented with a low recycling rate. According to a study conducted by United Nation University (UNU), e-waste is growing at an alarming compounded annual growth rate (CAGR) of 3–5%, and globally 53.6 million tons of e-waste is produced in 2019 and is anticipated to reach 74.7 million tons by 2030. Eco-friendly recovery of metals from e-waste is the need of the hour to protect the environment, conservation of natural resources, and resource re-utilization. Therefore, e-waste is considered a secondary resource, and recovery of metals from them is popularly known as “Urban Mining”. The existing popular routes for recycling metals from e-waste are mainly hydrometallurgical and pyrometallurgical processes and the metal recovery operations mostly take place in a multistep mode, which may include recovery of base metals, precious metals, or both. However, the generation of toxic effluents, high lixiviant consumption, hazardous emissions, and high energy consumptions are major hiccups in hydrometallurgical and pyrometallurgical processes.

Addressing the above challenges, this study focused on the development of an effective and environment-friendly method to extract valuable metals from waste printed circuit boards (PCBs) using low-temperature solid-state chlorination followed by the solvent leaching. Low-temperature roasting methods have several advantages over traditional hydrometallurgical and pyrometallurgical processes. High amount of metals can be recovered using this technique with high selectivity, low energy consumption, and low cost. In the present work, both chemical and thermal pre-treatment was used to process the polymeric fraction. Dimethyl formamide (DMF) was used in chemical pre-treatment to dissolve the

brominated epoxy resin (BER) from PCBs surface. More than 97% of the BER was separated using DMF at 140 °C. Similarly, through thermal pre-treatment, the polymeric fraction of the shredded PCBs was converted into oil and combustible gases. However, it was observed that the thermal pre-treatment method has more advantages compared to chemical pre-treatment. The yield of solid residuals through thermal pre-treatment was observed to be 77 wt.% solid residues, 5 wt.% liquid, and 18 wt.% gas under the optimized process condition of 500 °C temperature and 30 min of pyrolysis time. The metal-rich pyrolysis residue (T-PCBs) was roasted in presence of NH_4Cl as a chlorinating agent to recover metals. The effect of process parameters like temperature, NH_4Cl dose, and time was investigated to optimize the roasting process. Ammonium chloride at higher temperatures breaks into HCl and NH_3 , among which HCl reacts with metals to form metal chlorides. After completion of the chlorination reaction, the obtained residue was allowed to cool down and subsequently leached with a suitable solvent. Herein, water was used as a solvent for efficient extraction of base metals, whereas HCl solution was used for extraction of base as well as precious metals. Characterization techniques such as XRD and FESEM-EDS provided evidence for the successful conversion of metal into metal chlorides. This proposed method resulted in the extraction of 93% Cu, 100% Ni, 100% Zn, and 100% Pb using the water leaching technique when T-PCBs was chlorinated at 300 °C for 4 h with NH_4Cl dose of 3 g/g. Similarly, 98% Cu, 86% Ag, and 76% Au were extracted using HCl when T-PCBs was chlorinated at 260 °C for 2.5 h with NH_4Cl dose of 2 g/g. Ultimately, cementation methods were employed for individual separation of metals from leached solution. Further, a three-step cementation technique was employed for the separation of Au, Ag, and Cu using Ag, Cu, and Fe as sacrificial metals, respectively. Finally, all the reagents used in the process were recovered in the form of NH_4Cl and $\text{Fe}(\text{OH})_2$. Overall, this study demonstrated that low-temperature solid-state

chlorination could represent a promising approach for the recovery of metals from waste PCBs using a closed-loop process.

सारांश

सर्कुलर इकोनॉमी एक विकल्प नहीं बल्कि बढ़ती मांग और सीमित संसाधनों वाले हर क्षेत्र के लिए एक आवश्यकता है। इलेक्ट्रॉनिक कचरा (ई-कचरा) वर्तमान में पर्यावरण के लिए एक गंभीर खतरा पैदा करने वाले सबसे हानिकारक कचरे में से एक है, जिसमें मुख्य रूप से इसकी उच्च उत्पादन दर और कम रीसाइक्लिंग दर कारण है। यूनाइटेड नेशन यूनिवर्सिटी (UNU) द्वारा किए गए एक अध्ययन के अनुसार, ई-कचरा 3-5% की हानिकारक चक्रवृद्धि वार्षिक वृद्धि दर (CAGR) से बढ़ रहा है, और वैश्विक स्तर पर 2019 में 53.6 मिलियन टन ई-कचरे का उत्पादन हुआ है और इसका अनुमान है 2030 तक 74.7 मिलियन टन तक पहुंच जाएगा। पर्यावरण की रक्षा, प्राकृतिक संसाधनों के संरक्षण और संसाधनों के पुनः उपयोग के लिए ई-कचरे से धातुओं की पर्यावरण के अनुकूल पुनर्प्राप्ति समय की आवश्यकता है। इसलिए, ई-कचरे को एक द्वितीयक संसाधन माना जाता है, और उनसे धातुओं की प्राप्ति को "शहरी खनन" के रूप में जाना जाता है। ई-कचरे से धातुओं के पुनर्चक्रण के लिए मौजूदा लोकप्रिय मार्ग मुख्य रूप से हाइड्रोमेटालर्जिकल और पाइरोमेटालर्जिकल प्रक्रियाएं हैं और धातु पुनर्प्राप्ति प्रक्रिया ज्यादातर मल्टीस्टेप मोड में होते हैं, जिसमें आधार धातु, कीमती धातुओं या दोनों की पुनर्प्राप्ति शामिल हो सकती है। हालांकि, जहरीले अपशिष्टों का उत्पादन, उच्च तरल पदार्थ की खपत, खतरनाक उत्सर्जन और उच्च ऊर्जा की खपत हाइड्रोमेटालर्जिकल और पाइरोमेटालर्जिकल प्रक्रियाओं में प्रमुख व्यवधान हैं।

उपरोक्त चुनौतियों को संबोधित करते हुए, इस अध्ययन में कम तापमान वाले ठोस- पदार्थ क्लोरीनीकरण के बाद सॉल्वेंट लीचिंग का उपयोग करके अपशिष्ट मुद्रित सर्किट बोर्ड (पीसीबी) से मूल्यवान धातुओं को निकालने के लिए एक प्रभावी और पर्यावरण-अनुकूल विधि के विकास पर ध्यान केंद्रित किया है। पारंपरिक हाइड्रोमेटालर्जिकल और पाइरोमेटालर्जिकल प्रक्रियाओं की तुलना में कम तापमान वाली रोस्टिंग विधियों के कई फायदे हैं। इस तकनीक का उपयोग करके उच्च चयनात्मकता, कम ऊर्जा खपत और कम लागत के साथ धातुओं की उच्च मात्रा को पुनर्प्राप्त किया जा सकता है। वर्तमान कार्य में, बहुलक अंश को संसाधित करने के लिए रासायनिक और थर्मल पूर्व उपचार दोनों का उपयोग किया गया था। पीसीबी की सतह से ब्रोमिनेटेड एपॉक्सी राल (बीईआर) को भंग करने के लिए डाइमिथाइल फॉर्माइड (डीएमएफ) का उपयोग रासायनिक पूर्व-उपचार में किया गया था। 140 °C पर डीएमएफ का उपयोग करके 97% से अधिक बीईआर को अलग किया गया था।

इसी तरह, थर्मल पूर्व-उपचार के माध्यम से, पीसीबी के टुकड़े के पॉलीमर अंश को तेल और ज्वलनशील गैसों में परिवर्तित किया गया। हालांकि, यह देखा गया कि रासायनिक पूर्व-उपचार की तुलना में थर्मल पूर्व-उपचार पद्धति के अधिक फायदे हैं। थर्मल पूर्व-उपचार के माध्यम से

ठोस अवशेषों की उपज 500 °C तापमान और 30 min पायरोलिसिस समय की अनुकूलित प्रक्रिया स्थिति के तहत 77 wt.% ठोस अवशेष, 5 wt.% तरल और 18 wt.% गैस प्राप्त हुये । धातुओं को पुनर्प्राप्त करने के लिए क्लोरीनेटिंग एजेंट के रूप में NH_4Cl की उपस्थिति में धातु से भरपूर पायरोलिसिस अवशेष (T-PCBs) को रोस्टिंग किया गया था। रोस्टिंग प्रक्रिया को अनुकूलित करने के लिए तापमान, NH_4Cl मात्रा और समय जैसे प्रक्रिया-मापदंडों के प्रभाव की जांच की गई। उच्च तापमान पर NH_4Cl , HCl और NH_3 में टूट जाता है, जिसमें HCl धातुओं के साथ धातु क्लोराइड बनाने के लिए प्रतिक्रिया करता है। क्लोरीनीकरण प्रतिक्रिया के पूरा होने के बाद, प्राप्त अवशेषों को ठंडा होने दिया गया और बाद में एक उपयुक्त सॉल्वेंट के साथ निक्षालन किया गया। इसमें आधार धातुओं के कुशल निष्कर्षण के लिए पानी का उपयोग सॉल्वेंट के रूप में किया गया था, जबकि HCl लीचिंग का उपयोग आधार के साथ-साथ कीमती धातुओं के निष्कर्षण के लिए किया गया था। XRD और FESEM-EDS जैसी विशेषता तकनीकों ने धातु के क्लोराइड में धातु के सफल रूपांतरण के लिए साक्ष्य प्रदान किए। इस प्रस्तावित विधि के परिणामस्वरूप 93% Cu, 100% Ni, 100% Zn, और 100% Pb पानी की लीचिंग तकनीक का उपयोग करते हुए निकाला गया। जब T-PCBs को 300 °C पर 4 घंटे के लिए NH_4Cl की 3 g/g की खुराक के साथ क्लोरीनयुक्त किया गया। इसी तरह, 98% Cu, 86% Ag, और 76% Au को HCl का उपयोग करके निकाला गया। जब T-PCBs को 260 °C पर 2.5 h के लिए NH_4Cl की 2 g/g की खुराक के साथ क्लोरीनयुक्त किया गया। अंततः, निक्षालित सॉल्वेंट से धातुओं के अलग-अलग पृथक्करण के लिए सिमेंटेशन विधियों को नियोजित किया गया था। इसके अलावा, Au, Ag, और Cu को अलग करने के लिए क्रमशः Ag, Cu, और Fe को बलिदान-धातुओं के रूप में उपयोग करने के लिए एक तीन-चरण सिमेंटेशन तकनीक उपयोग की गई थी। अंत में, प्रक्रिया में प्रयुक्त सभी अभिकर्मकों को NH_4Cl और $\text{Fe}(\text{OH})_2$ के रूप में पुनर्प्राप्त किया गया। कुल मिलाकर, इस अध्ययन ने प्रदर्शित किया कि निम्न-तापमान ठोस-पदार्थ क्लोरीनीकरण एक बंद-लूप प्रक्रिया का उपयोग करके अपशिष्ट पीसीबी से धातुओं की पुनर्प्राप्ति के लिए एक आशाजनक दृष्टिकोण का प्रतिनिधित्व कर सकता है।

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