Recycling Printed Circuit Boards to Recover Valuable Metals

Grant Proposal

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Executive Summary

Efficient e-waste recycling becomes imperative while facing the increase in metal demand and decline in ore grades. This project proposes an effective recycling method for printed circuit boards (PCBs) to overcome the limitations of existing landfill disposal and pyrometallurgical methods, which are not only environmentally detrimental but also inefficient in resource recovery.

The proposed approach combines mechanical and hydrometallurgical techniques. This multiple-step process begins with a wet chemical dissolution process using acid to remove electronic components (ECs) from PCBs. Then, mechanical milling and dense medium separation are developed to extract copper from de-populated PCBs. Preliminary results show promising outcomes: nitric acid effectively removes ECs, while potassium hydroxide successfully strips solder masks, indicating a viable pathway for efficient metal recovery. The possible reuse and recycling of solutions involved in this multistep process will be investigated further to improve the environmental friendliness of the entire recycling process.

By developing a recycling process that requires lower energy inputs and produces fewer pollutants compared to traditional methods, this project aligns with global sustainability goals. The expected outcomes of the proposed recycling process include the recovery of valuable materials like copper foils, ECs, and various metal precipitates. These products have substantial market value and can be supplied to specialized manufacturing companies, thus meeting the metal demand of our society and developing a sustainable industry.

Keywords: e-waste, hydrometallurgy, sustainability, recycling, valuable metals,

Recycling Printed Circuit Boards to Recover Valuable Metals

The increasing demand for metals, driven by the rapid expansion of technology and electronics, highlights a critical challenge in resource management and environmental sustainability. At the broader level, this trend reflects a global shift towards greater reliance on technology, impacting not only resource consumption but also waste generation. The global demand for metals is expected to increase approximately 2–6-fold depending on the metal in the 21st century (Watari et al., 2021). In the meantime, the concentrations of useful minerals in ores have been and will invariably continue to decline (Mudd, 2007). These declining ore grades lead to an increase in solid wastes (tailings and waste rock) and energy consumption per unit of metal production. Thus, recovering valuable metals from waste streams becomes critically important to meeting demand and developing a sustainable industry.

E-waste is the fastest-growing waste stream in the United States and the world. According to a global study by the United Nations (UN), the world generated 53.6 Mt of e-waste in 2019 (Forti et al., 2020). This amount is projected to grow to 74.7 Mt by 2030. However, the recycling of e-waste is not challenging due to the complexity of materials used to construct electronic devices. Only 17. 4% of e-waste is formally collected and recycled globally (Forti et al., 2020).

Printed circuit boards (PCBs) are considered the most valuable e-waste for recycling because of their high content of valuable metals (Golev et al., 2019). In fact, in advanced consumer electronics, PCBs represent more than 80% of the total metal recovery value. Currently, PCBs are either disposed of in landfills or treated by pyrometallurgical methods (Zhu et al., 2023). However, the disposal of PCBs in landfills wastes valuable materials and can release harmful pollutants into the environment through leaching. Pyrometallurgy, i.e. burning PCBs at high temperatures for recovery of valuable metals, is energy-intensive, demands large capital investment, and requires the treatment of harmful gases and dust, such as dioxin or furan (Ahirwar & Tripathi, 2021).

To solve the problem, this project established a scalable, effective, and sustainable process to reclaim and recycle valuable metals from PCBs through a combination of mechanical and hydrometallurgical methods.

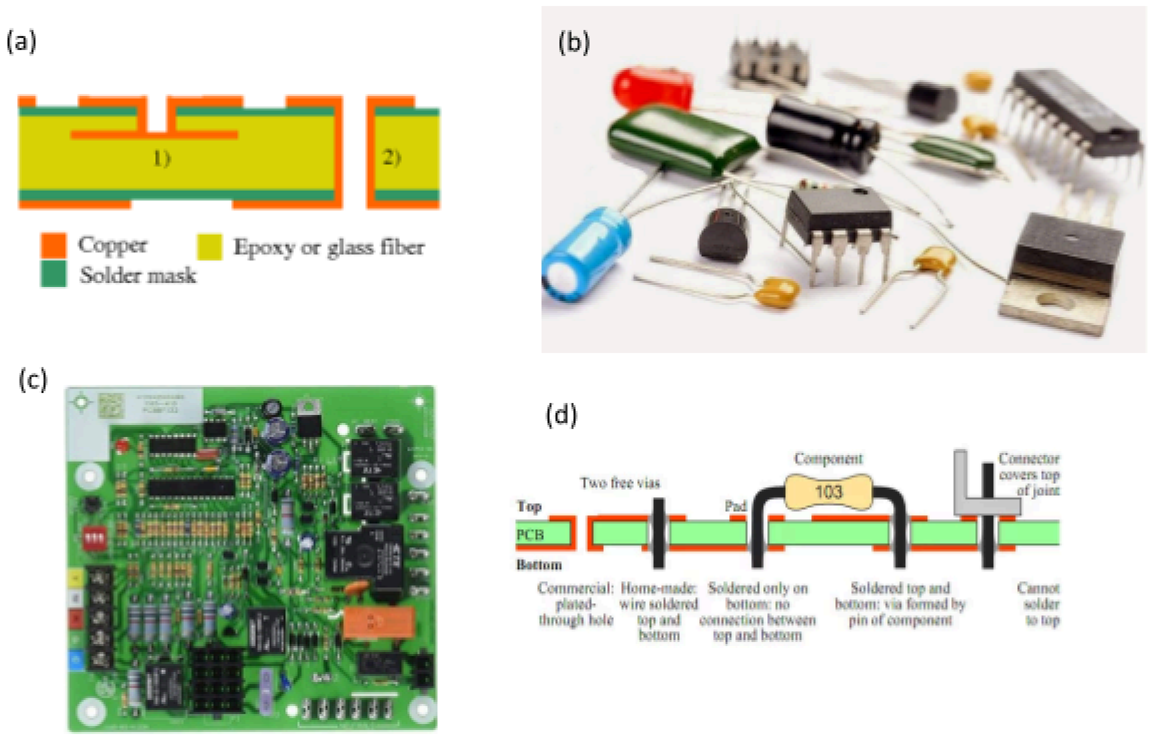

Figure 1. (a) Top view of a PCB example (The Goodman Store, n.d.). (b) Examples of major ECs commonly used in PCBs *(*The basics of PCB design, 2018). (c) Schematic cross-sessional view of a PWB (Tamhankar & Patel, 2012). (d) Schematic cross-sessional view of a part of a PCB with ECs (Ysadmin, 2015).

The effective recycling of waste PCBs is challenging due to their complexity. Typical PCBs consist of three parts: a plastic laminate substrate, a conducting metal layer on or inside the laminate, which is typically copper, and various electronic components (ECs) such as resistors, chips, and connectors that are mantled on the PCBs by soldering. The laminate substrate with the conducting metal layer is also referred to as a printed wiring board (PWB). Figure 1 (a) shows the top view of a PCB example. Figure 1 (b) shows examples of major ECs that are commonly used in PCBs. Figure 1 (c) shows a schematic of the cross-session view of a PWB that includes the epoxy laminate, the copper layers, and the solder mask.

Figure 1 (d) shows a schematic of the cross-session view of a part of a PCB that includes a resistor, vias, connectors, and solder that anchors ECs to the PWB.

To recycle PCBs, the first step is to remove the ECs and obtain unpopulated PCBs, or PWBs. In this project, a wet chemical dissolution method will be established to dismantle ECs from PCBs. The metal elements enriched in this step will be collected for refinement. After the EC removal process, a mechanical milling and dense medium separation combined treatment method for high-efficiency recovery of copper from PWBs will be developed. The possible reuse and recycling of the solutions involved in this multistep process will be investigated to further improve the environmental friendliness of the entire recycling process.

This project will combine mechanical and hydrometallurgical methods to recover valuable parts like ECs and metals, including Sn and Cu, from PCBs while minimizing the impact on the environment. This process requires low capital investment compared with pyrometallurgical methods and is economically and environmentally beneficial compared with pyrometallurgical methods and landfilling. The expected products of this process include ECs, precipitates such as $Cu(OH)_2$ and H_2SnO_3, and Cu foils. Each of these products is a valuable feeding stock material that can be sold to well-established specialized companies for manufacturing.

Section II: Specific Aims

The long-term goal of this project is to establish a scalable, effective, and sustainable process to reclaim and recycle valuable parts and metals from waste PCBs.

Rationale

Studies have shown that the concentrations of many metals in PCBs are much higher than in natural ores. For example, the concentration of Au in PCBs was found to be more than ten times its concentration in natural ore (Szałatkiewicz, 2014). Additionally, the concentration of copper in PCBs is 32 times higher than in the global average copper ore (Li et al., 2019). A recent study concluded that obtaining copper and gold from ores would cost seven times more than refining metals from PCBs (Zeng et al., 2018).

However, PCBs are considered the most intricate component of electrical products (Mughees, 2022). Only 28% of the metal content of the PCB waste is recovered by current recycling techniques. In addition, many current PCB recycling processes are not environmentally friendly options.

Thus, this engineering project will address the critical and urgent need to create a more efficient and sustainable recycling process for waste PCBs. We propose to develop a multiple-step process, including ECs dismantling, mechanical milling to separate plastic laminates and copper foils, dense medium separation to collect copper, and recycling of chemicals involved in each step of the process. The following specific aims will be addressed in this project to achieve this long-term goal.

Specific Aim 1: To develop an effective process to remove ECs from PCBs.

Specific Aim 2: To develop an effective process to reclaim and recycle valuable metals from unpopulated PCBs.

Specific Aim 3: To improve the environmental friendliness of the developed process by studying the effective reuse of the chemicals involved.

Specific Aim 4: To understand the enrichment of metal elements in chemical solvents throughout the process and to effectively collect the metal elements from solvents at the end of the process.

The expected outcome of this work is an efficient process to reclaim valuable parts and metals from waste PCBs that can be transferred to the industry for commercialization.

Section III: Project Goals and Methodology

Relevance/Significance

Table 1 shows the typical composition of metals in PCBs. It shows that PCBs contain many high-value metals, such as copper, iron, tin, and gold, and many hazardous substances, like lead. A study estimated that the metal value in waste PCBs in Australia was at US$ 150 million a year. However, less than a third of this value is captured in a recent Australian study (Golev & Corder, 2017). The Australian study is a good indicator that the efficient recycling of waste PCBs is needed for both economic value and protection of the environment.

Table 1. Range of metal composition of PCBs in the literature (Van Yken et al., 2021)

Element	Composition (%)	Element	Composition (ppm)
Cu	6–40	Au	250–2050
Fe	1.2–8	Ag	110–4500
Al	0.3–7.2	Pd	50–4000
Sn	1–6.3	Pt	5–30
Pb	1–4.2	Co	1–4000
Ni	0.0024–5.4	-	-
Zn	0.04–2.2	-	-
Sb	0.1–0.4	-	-

Innovation

Despite the high value of metal contents, PCBs are recycled at a low rate due to the low efficiency of manual dismantling and pyrometallurgical recycling methods. The proposed process will combine hydrometallurgical and mechanical methods to create a more efficient and sustainable process for recycling PCBs.

Methodology

Specific Aim 1: To develop an effective process to remove ECs from PCBs

Research to dismantle ECs has received much attention in the European Union and China but not in the United States (Maurice et al., 2021). Effective dismantling ECs from PCBs is critical to increasing recovery yields and creating processes suitable for small-volume production with low capital investment. Table 2 summarizes various EC dismantling methods, comparing their relative costs in terms of operating expenses (OPEX) and capital expenses (CAPEX), as well as their advantages and drawbacks (Maurice et al., 2021). Currently, ECs are removed manually, making it a labor-intensive, costly, yet necessary process. Thermal processes that heat the PCBs to 40 and 50 °C higher than the melting temperature of the solder have been studied; however, these studies have shown that the release of toxic substances such as dioxin and related compounds during thermal processing is a concern (Duan et al., 2011; Zhang et al., 2015). Thermal methods also require significant capital investment and high energy consumption. In addition, thermal treatment may damage ECs that could be reused (Maurice et al., 2021). Therefore, a wet dismantling process at a low temperature that requires low capital cost is an attractive alternative.

In this project, a hydrometallurgy method to produce unpopulated PCBs will be developed. This method is easily scalable, efficient, and cost-effective. Moreover, the tin and other metal elements are enriched during this process and can be sold to the industry as feeding materials for well-established commercial metal refining processes. Another potential advantage of this approach is remanufacturing of ECs for reuse. The collected ECs can be sold at relatively high prices to precious metal processing companies (Electronic scrap, n.d.) or remanufactured for continued service. Thus, developing a wet dismantling process compatible with the current PCB manufacturing process is desirable. Such a process could better enable the remanufacturing of ECs compared with thermal and mechanical dismantling processes (Stennett & Whalley, 1999).

Table 2. Qualitative comparison of the different dismantling processes (Maurice et al., 2021).

Process	CAPEX	OPEX	Advantages	Drawbacks
Manual dismantling	+	++++	Easy to implement Selective disassembling	Hard manual work Requires manpower Slow process Polluting
Surface cutting knife	++	+	Non-polluting High disassembly rate	WPCBs are treated one by one
Crude heating	++	+	Large capacity	Toxic emissions Heat damage ECs
Infrared radiators	++	+	High disassembly rate	Small volumes Heat damage ECs
Hot air heating	++	++	High disassembly rate Little maintenance Non-polluting	Low accuracy control Low energy efficiency
Solder bath heating	++	+++	High disassembly rate	Difficult to automate Toxic fumes emissions Dangerous working conditions
Hot fluid heating	++	++	High disassembly rate High thermal efficiency High solder recovery rate	Generating toxic waste fluids and fumes
Heated centrifugation	++	++	High solder recovery rate Solder elements separation	High temperature Heat damage ECs
Solder dissolution	+	++	Selective process	Requires further treatments Hazardous chemicals used
Hydrothermal and supercritical fluids treatment	+++	++	Target metals or resin No toxic product released Reusable reagents	Cannot target solder Requires further dismantling treatments
Epoxy resin treatment	++	++	Recover functional circuits Chemicals can be recycled	Need further dismantling process Hazardous chemicals used
Robotic Dismantling	+++	+	Combined dismantling and sorting Low manpower	Low throughput
Fragmentation by high voltage electric pulse crusher	+++	++++	High capacity Non-polluting	Low energy efficiency Expensive initial investment

Note: + cheap, ++ mid-priced, +++ expensive

Various solvents for chemical dissolution to dismantle ECs have been studied, including nitric acid (Stennett & Whalley, 1999; Yang et al., 2017), hydrochloric acid (Jung et al., 2017), ionic liquids (Zeng

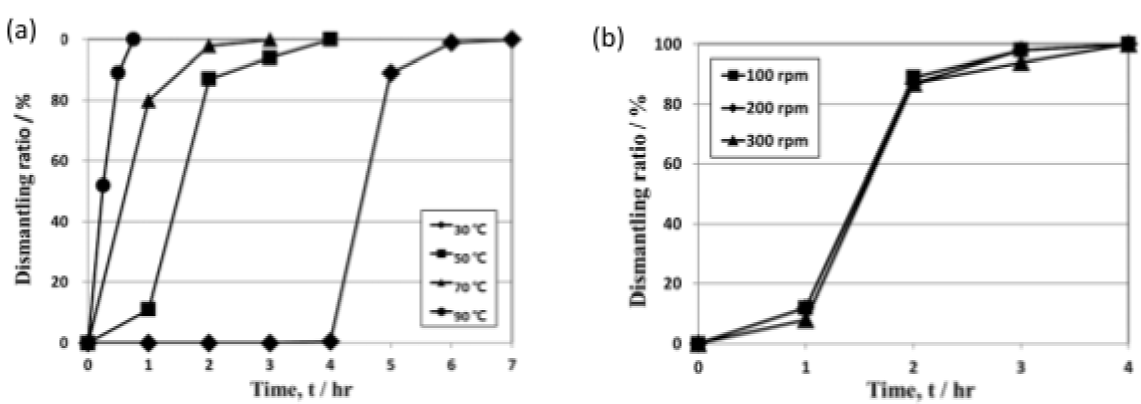

Figure 2. (a) Dismantling ratio in the temperature range from 30–90°C in 1 mol/L HCl with 10,000 mg/L Sn4+ at 300 rpm. (b)The effect of agitation speeds on the dismantling ratio in 1 mol/L HCl with 10,000 mg/L Sn4+ at 50°C and 100–300 rpm (Jung et al., 2017).

et al., 2013), fluoroboric acid with hydrogen peroxide (Zhang et al., 2015), and methanesulfonic acid (Zhang et al., 2017) with hydrogen peroxide. However, the high cost of ionic liquid and the instability of hydrogen peroxide hinder commercialization. Thus, in this project, nitric acid and hydrochloric acid-based recipes will be studied. The variables that will be studied include acid concentrations, the effect of additives, agitation, and reaction temperatures. The selection of the ranges of these variables to be tested will be informed by similar previous studies and the costs. For example, the range for agitation speed for hydrochloric acid-based solutions will be from no agitation to 300 rpm, and the temperature range will be from room temperature to 90°C, informed by the results shown in Figure 2.

This project will study the reuse of the solutions and the treatment of the spent solutions for safe disposal in Specific Aim 3 to alleviate the environmental impact of the used solvents,

The overall outcome is a cost-effective and efficient recipe to remove ECs.

Summary of Preliminary Data

A preliminary experiment was conducted to test the impact of nitric acid concentration on the EC dismantling speed by wet chemical dissolution. Tang et al. (2021) proposed a common pickling solution to remove Sn and Pb from PCBs. This was employed as a starting point and is comprised of 40 vol% nitric acid (HNO3), 40 g/L ferric nitrate (Fe(NO3)3), 5 g/L ferric chloride (FeCl3), 5 g/L sodium chloride (NaCl), 10 g/L benzotriazole (C6H5N3), and 5 g/L sulfamic acid (NH2SO3H). Nitric acid reacts with Sn, Pb, Cu, and other metals according to the following reactions:

$$3Sn + 4HNO_3 + H_2O \rightarrow 3H_2SnO_3\downarrow + 4NO\uparrow \qquad (1)$$

$$Sn + 4HNO_3 \rightarrow H_2SnO_3\downarrow + 4NO_2\uparrow + H_2O \qquad (2)$$

$$4Sn + 10HNO_3 \rightarrow NH_4NO_3 + 4Sn(NO_3)_2 + 3H_2O \qquad (3)$$

$$3Pb + 8HNO_3 \rightarrow 3Pb(NO_3)_2 + 2NO\uparrow + 4H_2O \qquad (4)$$

$$3Cu + 8HNO_3 \rightarrow 3Cu(NO_3)_2 + 2NO\uparrow + 4H_2O \quad (5)$$

Additives, including ferric nitrate, ferric chloride, sodium chloride, benzotriazole, and sulfamic acid, were used to remove the underlying tin-copper alloy and improve the insulation resistance of the printed circuit board (Johnson & Fakler, 2001).

In this preliminary experiment, the concentration of nitric acid was varied from 20 vol% to 40 vol% at a step of 5 vol%. Composition of additives were kept constant in each solution at 40 g/L ferric nitrate (Fe(NO3)3), 5 g/L ferric chloride (FeCl3), 5 g/L sodium chloride (NaCl), 10 g/L benzotriazole (C6H5N3), and 5 g/L sulfamic acid (NH2SO3H). 3 ml of each solution was used. Waste PCBs were cut into small pieces with ECs on each piece and weighed. The experiment was carried out using a 1 g PCB: 3 ml solution ratio for each solution. The experiment was conducted at room temperature for 90 minutes. Table 3 summarizes the initial weight of PCB samples, the corresponding nitric acid concentration, the observed outcomes, and the weight of unpopulated PCBs after EC removal. Figure 3 shows the color of the solutions at the end of the experiment. It is evident that the higher the nitric acid concentration, the bluer the solution at the end. The blue color signifies the dissolution of copper. 40 vol% resulted in the fastest EC removal. Concentration lower than 35 vol% did not result in complete EC removal after 90 minutes. 35 vol% resulted in completed EC removal in 70 minutes. Thus, the preliminary results strongly support the conclusion that the concentration of nitric acid plays a significant role in the speed of EC removal.

Table 3. Impact of nitric acid concentration on EC removal

PCB start weight (g)	HNO_3 concentration	time	PCB end weight (g)
1.09	40 vol%	38 mins	0.93
0.739	35 vol%	70 mins	0.60
0.839	30 vol%	Did not fully remove EC after 90 minute	N/A
0.839	25 vol%	Did not fully remove EC after 90 minute	N/A
0.709	20 vol%	Did not fully remove EC after 90 minute	N/A

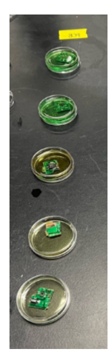

Figure 3. Impact of nitric acid concentration on EC removal. From top to bottom: 40%, 35%, 30%, 25% and 20%.

Specific Aim 2: To develop an effective process to reclaim and recycle valuable metals from unpopulated PCBs

Unpopulated PCBs, PWBs, are non-conducting laminates with conducting circuits, typically made with thin copper foil, printed in or on them (Figure 1 (c)). To protect the copper from oxidation and shorting during operation, a protective thin epoxy layer, known as a solder mask, is put on the board (Figure 1 (c)). To recover the copper from PWBs by mechanical methods, this solder mask layer should be first removed to avoid forming a black coating on the copper surface after the energetic mechanical processing. This black coating makes characterization and subsequent copper refining more challenging. Sulfuric acid (Imre-Lucaci et al., 2012), sodium hydroxide (Jadhav & Hocheng, 2015), and laser (Raele et al., 2017) have been studied to remove the solder mask. Sodium hydroxide is effective in removing solder masks of different epoxy resin compositions (Balaji et al., 2021). It was shown that at room temperature and atmospheric pressure, the solder mask could be completely removed by soaking the waste PCB in 5M NaOH for 8 hours and followed by sonication for 5 minutes (Senophiyah-Mary et al., 2018). In this project, NaOH and KOH will be studied for removing solder masks. The reuse and safe disposal of the solder mask removal solution will also be considered.

After the solder mask removal, the PWBs will be shredded by a mechanical miller. It is hypothesized that during the milling process, delamination will result in the separation of Cu foil from the laminate substrate. Due to the density difference in Cu (8.96 g/cm^3) and laminates (< 2g/cm3) (Tuncuk et al., 2012), a density separation method can be developed to collect Cu foils and plastic laminates effectively. A bromoform and acetone mixture solution has been shown to be effective in separating metal-enriched pieces and metal-depleted pieces after milling of waste PCBs (with ECs) (Nekouei et al., 2018). However, due to the concern of bromoform being more hazardous with higher vapor pressure (5.9 mm Hg at 25°C) compared with tetrabromoethane (TBE) (Hauff & Airey, 1980), TBE will be used as the starting point of density separation. TBE has a density of 2.95 g/mL and a low vapor pressure (0.02 mmHg at 25°C). It is widely used in mineral separations.

The overall outcomes include an effective recipe to remove the solder mask, efficient mechanical milling, and a density separation protocol to collect copper foils that can be readily used as feeding materials in well-established copper refining processes.

Summary of Preliminary Data

Previous studies show that solder masks can be removed by 3M NaOH at 70 °C for 2 h (Kang et al., 2021). To test if KOH would work similarly, a preliminary experiment was conducted using 2M KOH

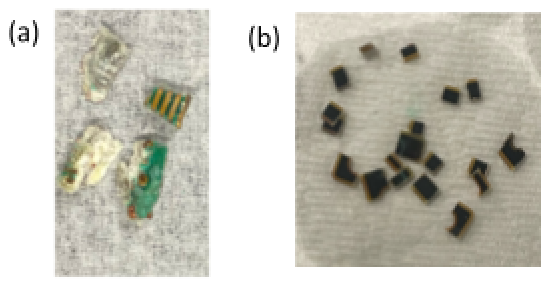

Figure 4. (a) PCBs treated with 2M NaOH at 90°C for 90 min. (b) PCBs treated with 2M KOH at 90°C for 90 min.

and 2M NaOH at 90°C for 90 min at a solid-to-liquid ratio of 1g:5ml. Figure 4 (a) shows the samples treated with 2M NaOH, and Figure 4 (b) shows the samples treated with 2M KOH. It is evident that at the end of the experiment, the solder mask remained on the samples treated with 2M NaOH while the solder mask was fully removed from the samples treated with 2M KOH. Although KOH is about 30% more expensive than NaOH (NaOH vs KOH, 2015), being more effective in removing the solder mask at a lower concentration can offset this price difference. Thus, it is worthwhile to further compare the effectiveness of KOH in removing solder masks.

Specific Aim 3: To improve the environmental friendliness of the developed process by studying the effective reuse of the chemicals involved

Several steps in the proposed process use solutions, with these solutions being an acid-based solution to dismantle ECs, an alkali solution to remove the solder mask, and a dense medium solution to separate copper from plastic laminates. The reuse of these solutions will be studied in this project. At the end of each reaction, used solutions will be filtered to obtain clean used solutions. Then, fresh solutions will be added only to maintain constant volume-to-volume or volume-to-weight ratios with the waste pieces to be treated. The effectiveness of the used solutions to treat waste pieces will be observed and documented to establish the protocols for reusing these solutions.

The expected outcome is the protocols to reuse and safely dispose of chemicals involved in the process.

Specific Aim 4: To understand the enrichment of metal elements in chemical solvents throughout the process and to effectively collect the metal elements from solvents at the end of the process

The acid solution used to remove solder is likely to dissolve other valuable metal elements, including Cu, Fe, Pb, Ni, and Zn (Table 1). Thus, the concentration of these elements in the fresh solution and the spent solution (at the end of the process) will be measured by ICP-OES to understand the enrichment of metal elements in the solution. Potassium hydroxide can effectively precipitate these metal elements according to the following reactions:

$$Cu^{2+} + 2OH^- \rightarrow Cu(OH)_2\downarrow \quad (6)$$

$$Sn^{4+} + 4OH^- \rightarrow Sn(OH)_4\downarrow \quad (7)$$

$$Fe^{3+} + 3OH^- \rightarrow Fe(OH)_3\downarrow \quad (8)$$

$$Ni^{2+} + 2OH^- \rightarrow Ni(OH)_2\downarrow \quad (9)$$

$$Zn^{2+} + 2OH^- \rightarrow Zn(OH)_2\downarrow \quad (10)$$

$$Pb^{2+} + 2OH^- \rightarrow Pb(OH)_2\downarrow \quad (11)$$

Potassium hydroxide will be added to the spent solution at the end of the process. Centrifuging will be used to collect the precipitates. Energy-dispersive X-ray (EDX) will be used to analyze the composition of the precipitates, and inductively coupled plasma-optical emission spectrometry (ICP-OES) will be used to confirm the complete precipitation of the metal elements from the spent solution at the end.

Similar studies will be conducted for the other two solutions used in the process.

The expected outcome is the effective reclaim of all the valuable metal elements from the spent solutions at the end of the process.

Figure 5 summarizes the design of the proposed process.

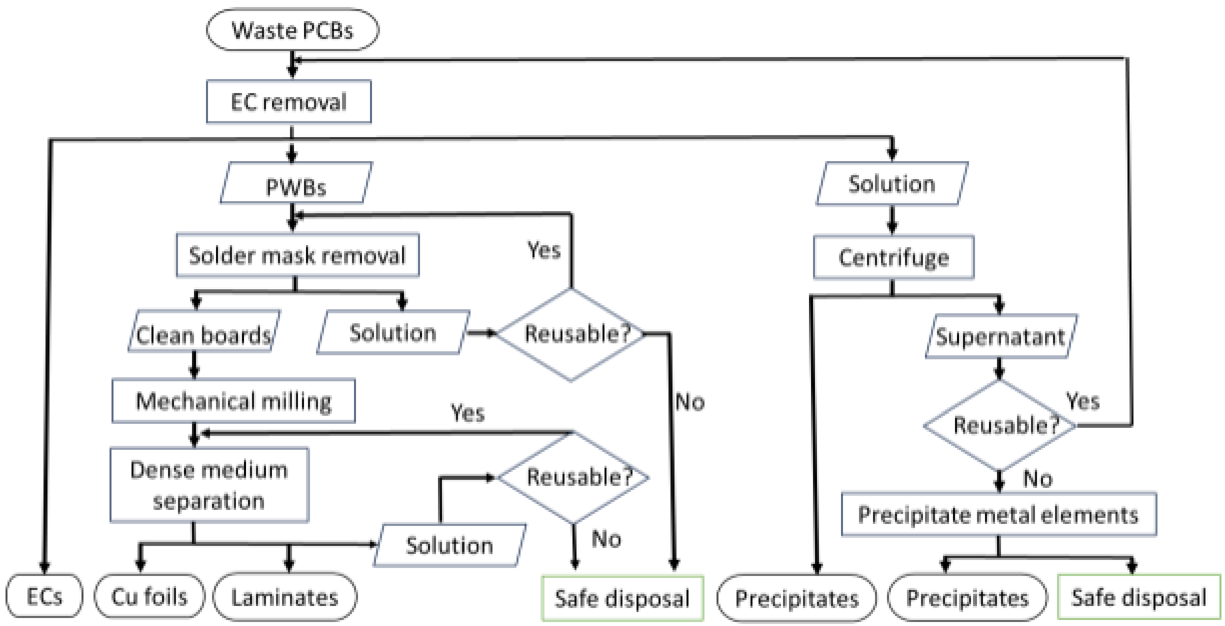

Figure 5. The proposed process. (Note: filtration that will be used to separate solutions from solids several times in this process is not shown on this schematic flow diagram to make the illustration easy to follow.)

Section IV: Resources/Equipment

Materials needed to carry out the proposed work include:

- waste PCBs
- chemicals: nitric acid, hydrochloric acid, sulfuric acid, sodium hydroxide, potassium hydroxide, tetrabromoethane (TBE), acetone, and additives including ferric nitrate, ferric chloride, sodium chloride, benzotriazole, and sulfamic acid

Equipment needed for this project include:

- processing equipment: mechanical miller, furnace, centrifuge, filtration set, ultrasonic cleaner, magnetic stirrer, sieve, rotary cutter and balance
- characterization equipment: ICP-OES and EDX

Section V: Ethical Considerations

The proposed work involves the handling of hazardous acids and bases. In addition, toxic substances may exist in e-waste, like lead, mercury, and cadmium, that can pose health risks. Thus, health and safety are important considerations. Ensuring safe working conditions and proper protective equipment is essential.

Improper recycling methods can lead to environmental contamination. It's important to ensure that the developed recycling process minimizes environmental harm, such as prioritizing preventing the release of toxic substances into the air, water, and soil. This project will study the possible reuse of the chemicals and solutions involved in the proposed process to minimize environmental contamination.

Section VI: Timeline

Table 4. Timeline of the proposed project

	23-Sep	23-Oct	23-Nov	23-Dec	24-Jan	24-Feb
Brainstorming and idea generating	■					
Literature and background research	■	■				
Experiment setup and preperation		■	■			
EC removal			■	■		
Develop a process to recycle unpopulated PCBs			■	■		
Study the reuse of the chemicals involved			■	■	■	
Collect elements from spent solvents			■	■	■	
Data analysis			■	■	■	
Report					■	■

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