

Evaluating pre-treatment methods to maximise copper recovery from printed circuit boards

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Abstract : Waste Printed Circuit Boards (PCBs) are a valuable-metals rich fraction of the electronic waste (e-waste) stream. Copper is the most abundant metal on the PCBs, found on the surface of the boards, on the vias that connect copper layers and on the inner copper layers in the now common, but more complex, multilayered PCBs. Liberation of the inner copper layers presents significant challenges in the hydrometallurgical processing of the PCBs due to the impervious tight lamination of the multilayers, rendering lixiviant-metal contact impossible. The inaccessible copper is found in alternating layers of copper sheets and glass- reinforced epoxy laminate. Current practice employs smelting processes and/or energy extensive fine grinding to liberate the copper. This has the disadvantages of high energy costs, loss of materials in the size reduction steps and the potential emission of toxic gases if furnaces are not equipped with advanced gas scrubbers. This study investigates and compares the effectiveness of mechanical, e.g. shredding or cutting the PCBs, as well as chemical pre-treatment methods, e.g. organic solvent swelling or sodium hydroxide soaking, as well as an integration of the chemical and mechanical pre-treatment methods. The study employs ammonia-based copper leaching to evaluate the effectiveness of each pre-treatment method, using copper extractions as a proxy for assessing effective copper liberation in the precursor step. The study finds that a combination of pre-treatment steps yields very promising results, offering further opportunities using higher concentration and temperatures to achieve competitive rates for developing small-scale processes.

Keywords : Recycling, PCBs, Pre-treatment, Copper-leaching

1. INTRODUCTION :

Printed Circuit Boards (PCBs) are at the core of the electronics industry and are found in all Electrical and Electronic Equipment (EEE) [1]. Thus, when electrical and electronic equipment (EEE) reaches its end of use or end of life, PCBs become one of the fractions obtained following the dismantling and sorting of waste EEE, also called electronic waste (e-waste). Owing to the fast global consumption rate and hence high obsolescence rate of EEE, e-waste is the fastest growing waste stream in the world. With that, is the increased concern of pollution from this waste stream, yet still there are opportunities to recover valuable materials and contribute to a waste economy. Waste PCBs comprise of 3 - 10 wt % of the total electronic waste stream [2, 3, 4] and constitute up to 80% of the recovered value of the waste stream [3]. This value is due to the relatively high

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metal content, with the overall board made up of an estimated 40% metal [5]. Due to the variability in PCB uses and hence design of the board, copper content has been reported at varied concentrations, some of which are shown in Table 1. Notably, these copper grades are higher than those of primary copper ores indicated in recent reports which are in the range 0.3% to 2% [6,7]. And hence, beyond recycling as a means to contain the detrimental environmental impact of poor disposal of PCBs, there are also monetary gains and opportunities for the growth of a waste economy.

Table 1. Copper content on various types of boards.

| PCB Type | Copper Content | References |
|---------------------------|----------------|------------|
| Waste computer mainboards | 20% | [8] |
| TV boards | 10% | [9] |
| Generalized PCBs | 26% | [10] |
| Waste computer mainboards | 25.3% | [11] |
| Printers | 32.5% | [12] |
| Mobile Phones | 33.5% | [13] |

Copper is not the only valuable metal found in PCBs, with the highest monetary value being attributed to precious metals such as gold which is found at a range of concentrations (68 g/t to 1120 g/t, [14]) in the waste PCB stream. However, this paper focusses on copper which is the most abundant metal in the waste PCB stream.

A typical approach to the processing of PCBs to recover various metals is through smelting in large integrated pyro-hydrometallurgical plants which co-process various metals rich feed streams. Such operations harness scales of economy fed by the relatively more established e-waste collection systems and higher consumer patterns of electronics in developed countries, which in due course translates to higher volumes of e-waste [15]. Countries with developing economies suffer poor e-waste collection, relatively lower generation of e-waste, high energy costs and poor supporting regulations. Under those circumstances, it is uneconomical to run recycling facilities such as those found in developed nations, nor is it feasible to operationalize the technologies employed in the first world. Therefore, alternative processing routes, which are scalable and accessible to the small-medium scale recyclers are required to provide opportunities for full participation in the recycling economy. Hydrometallurgical processing routes have potential for application in the developing economies context. However, for hydrometallurgical processes to be effectively used to recover metal from waste PCBs, there needs to be effective contact between the desired metals and the leaching agents. This metal-lixiviant contact can be easily achieved for metals such as tin, lead, gold, nickel and other precious metals that sits on the surface of the PCB [16]. Copper, present in the highest concentrations on the board, is not as readily available, owing to the increasingly more complex structure and design of PCBs.

PCBs can be classified as single sided, double sided or multi-layered based on the number of conductive layers as illustrated in Figure 1. Structurally, PCBs are tightly laminated metal-polymer composites, whose metal and polymer layers retain the individual chemical, physical and mechanical properties. The polymer layers vary in material but in general comprise of tightly woven fiberglass embedded in epoxy. The fiberglass “provides mechanical strength and stiffness to the composite material due to the higher tensile strength and elastic modulus” [17]. Additives such as flame retardants, curing agents, fillers and accelerators are added to the resin as required for the specific function of the PCB [18]. The material is impervious, while also difficult to crush and mill. Wang et al [19] reported on the difficulty to crush PCBs using some of the common equipment in the comminution industry such as the jaw crusher, the roller crusher and the disk crusher, and recommended that crushers with cutting or impacting effect such as the hammer mill be used.

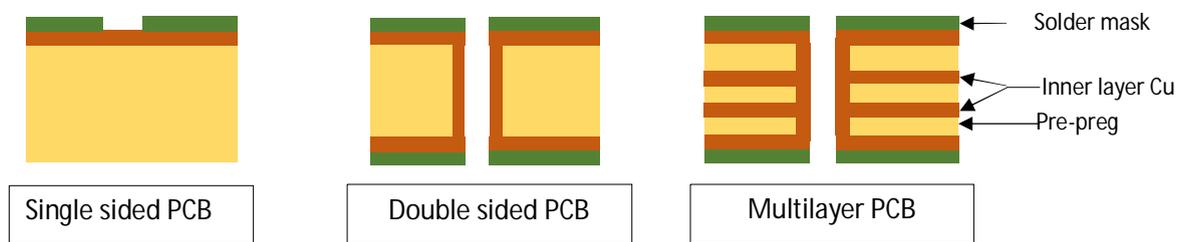


Figure 1. PCB classification

Due to the tight lamination between the metal and polymer-based materials, effective leaching requires total liberation of the metal fractions. This translates to high energy costs and thus various researchers have explored alternative methods to liberate metal from waste PCBs [20 - 25]. While these authors did some extensive test work to assess the liberation of metals, they neglected to link this to the resultant metal extractions in the leaching stage or only treated single and double layered PCBs. Adhasure et al [21] found NaOH to be effective in removing the solder mask coating on PCBs, thereby exposing all the outer laying copper, availing it for leaching. Researchers have reported positive results when testing organic solvents such as N-methyl-2-pyrrolidone (NMP), dimethyl sulfoxide (DMSO), di-methyl formamide (DMF) and Dimethyl acetamide (DMAc) [20, 22, 25]. These type organic solvents are effective for dissolving BER, the main constituent of the epoxy resin found in PCB laminates and 'pre-preg', which refers to the non-metallic polymer acting as a glue between the inner-layer copper and the laminates. The dissolution of the BER leads to the delamination of the polymer and metal, thereby exposing the metal and availing it for contact with leaching agents. Organic solvents have the advantage that they can be regenerated and recycled in the process [26, 27].

The aim of this study is to evaluate the effectiveness of different approaches taken to pre-treat PCBs in an effort to liberate copper and maximise its extraction. Mechanical and chemical pre-treatment methods, as well as an integration of the two will be explored.

2. MATERIALS AND METHODS

2.1. Printed Circuit Boards

Custom-made and uniformly designed PCBs (142 x 102 x 1.55 mm, shown in Figure 2), manufactured by a local PCB manufacturer (TraX Interconnect (Pty) Ltd.) were used in all experiments to overcome the heterogeneity of waste PCB streams and thus effectively compare results across the experiments. The PCB sample has 4 layers of Cu as shown in Figure 3. The head grade of the sample, i.e. copper content per PCB, was determined via shredding an individual PCB, pulverizing it, followed by acid digestion in aqua regia and reverse aqua regia. The solutions were analysed using ICP-MS, and the metal content was found to be 11,77 grams/ PCB. Each PCB was found to weigh $52,2 \pm 0.06$ grams in total.

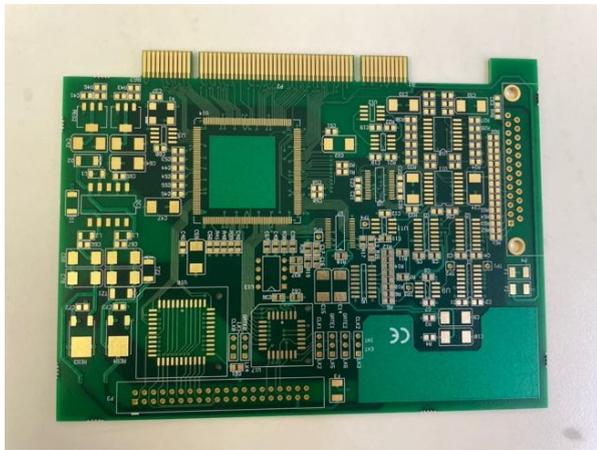


Figure 2a. Custom made PCB top surface layer

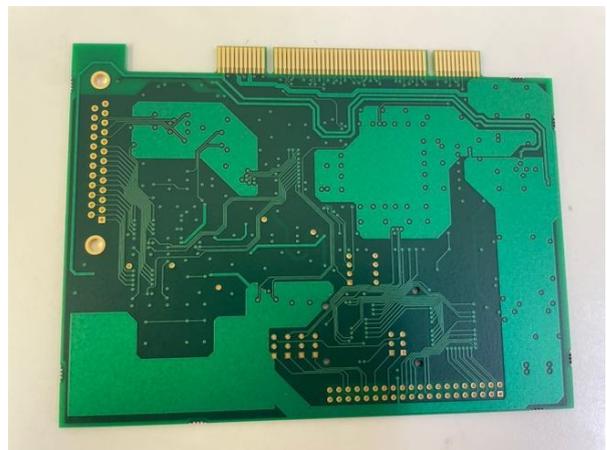


Figure 2b. Custom made PCB bottom surface layer



Figure 3. Cross-sectional view of the custom-made PCB (reproduced from [14])

One whole PCB designed in this fashion was used in each test of the different pre-treatment methods as described below, thus ensuring high repeatability of the results. After the various pre-treatment steps, the resultant material was washed and dried before leaching in a diagnostic leach experiment.

2.2. Pre-treatment methods

Two different pre-treatment methods were systematically reported in this study, shredding in an industrial grab shredder and softening/delaminating the PC Board material in organic solvents. A set of experiments was conducted to evaluate the efficacy of each method and refinement of the operating condition, followed by a comparison between the two approaches and their combination.

Shredder experiments: The principle of the shredder (Filamaker GmbH, Germany) and a picture are shown in Figure 4. Individual PC boards were fed through the shredder and the product collected in a tray, before being fed again, up to 6 cycles. The power consumption of the motor driving the shredder was recorded for each shredding cycle. The particle size distribution of the shredder product was determined by dry sieving on a sieve stack.

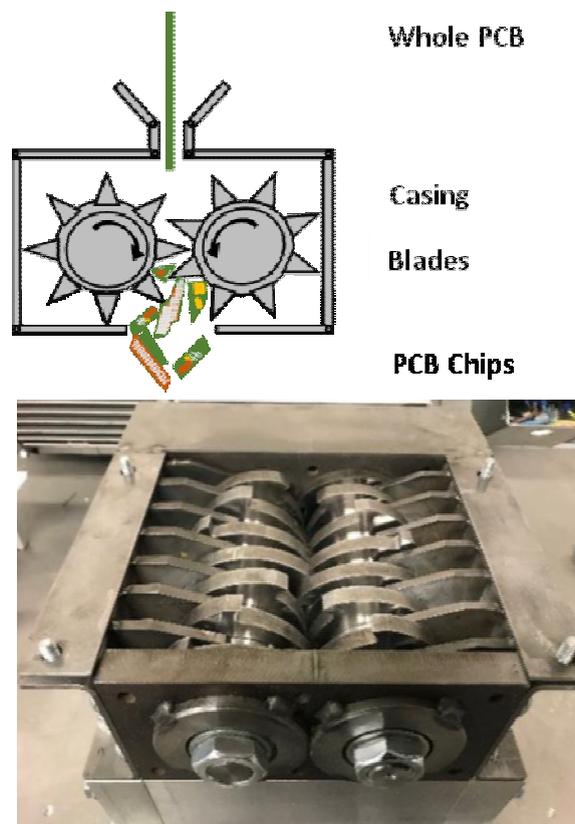


Figure 4. Principle of grab shredder and picture of the device (safety cover removed)

Organic swelling experiments: 3 organic swelling agents (were initially tested against each other using shredded PC boards under otherwise identical conditions in terms of length and temperature of the soaking reaction. The best performing agent (in terms of subsequent copper leaching) was subsequently optimized in terms of the temperature and length of the swelling reaction. The reagents tested were Dimethyl sulfoxide (DMSO), N-methyl-2-pyrrolidone (NMP) and a proprietary industrial de-smearing agent used in the

manufacture of PCBs, 'E-prep', which typically contains pyrrolidone compounds similar to NMP [28]. DMSO and NMP were supplied by Sigma, whereas the 'E-prep' is supplied by MacDermid Enthone.

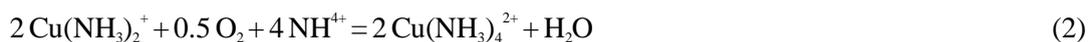
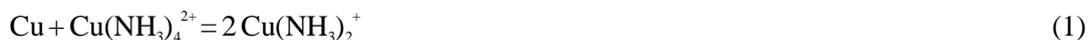
Comparative tests: 4 tests were conducted comparing leaching a cut untreated PCB, a cut PCB soaked in NMP, a shredded PCB and finally a shredded PCB subsequently soaked in NMP – the experimental details are given in Table 2.

Table 2. Pre-treatment methods used to liberate Cu from PCB samples.

| Method | Description |
|--|--|
| Method 1: Cut PCBs | An individual PCB was cut into 2x2 cm pieces for the purposes of fitting into a reactor. Preliminary tests indicated that cutting to this size alone did not result in improved leaching of Cu, as even the Cu on the edges of the PCB remained inaccessible when there were no further interventions. |
| Method 2: Shredding using an industrial grab shredder | An individual (whole) PCB was passed through an industrial grab shredder. The material went through 4 passes i.e. the material would be collected at the bottom of the shredder then re-fed into the shredder. |
| Method 3: Organic swelling of cut PCBs | In each experiment, an individual PCB was cut into 2X2 cm pieces (for the purpose of fitting into the reactor) and swelling was done in N-Methyl-2-pyrrolidone (NMP) at a solid to liquid ration of 1:5 for 90 min at 100 °C. |
| Method 4: Organic swelling of shredded PCBs | In each experiment, an individual PCB was passed through the shredder 4 times (as described in method 1). The shredded PCB pieces were then swollen in NMP at a solid to liquid ration of 1:5 for 90 min at 100 °C. |

2.3. Diagnostic leach tests

The leaching conditions used in this study were those as optimized by Chirume [16]. The key chemistry in Eqn. (1) and (2) is determined by the dissolution of copper and regeneration of cuprous through atmospheric oxygen.



In all leach experiments, in a lixiviant made up of 2 M (NH₄)₂CO₃, 4 M NH₄OH and 100 ppm initial CuSO₄ was used. Differences came primarily from different reactor systems used as the study evolved:

- ❖ The diagnostic leaches for the shredder tests were conducted in jacketed glass reactors maintained at 25°C and agitated by an overhead stirrer running at 100 rpm (the particles were largely not suspended). Air was bubbled into the reactor at a rate of 100 mL/min

- ❖ Tests for the swelling agent were conducted in a sealed bottle roller at ambient temperature (22–25°C). The sample was shredded in 4 cycles prior to swelling. As per Eqn. (2), oxygen contained in the bottle may become consumed in the course of the leach reaction.
- ❖ Combined tests were also carried out in bottle-rollers, but these were aerated through a gas fed pipe inserted in the lid (see Figure 5). Air was fed at 100 mL/min to each reactor. Experiments ran at ambient temperature (22–25°C).

The solid to liquid ratio was at 1:10 in effort to eliminate mass transfer limitations. The pH of the solutions was monitored and remained in the desired range of 9.6 – 10.2 across all reactors through the duration of each test. Solution samples were analysed using ICP-MS and dissolved copper normalized against the Cu head grade of the board (11.77g).

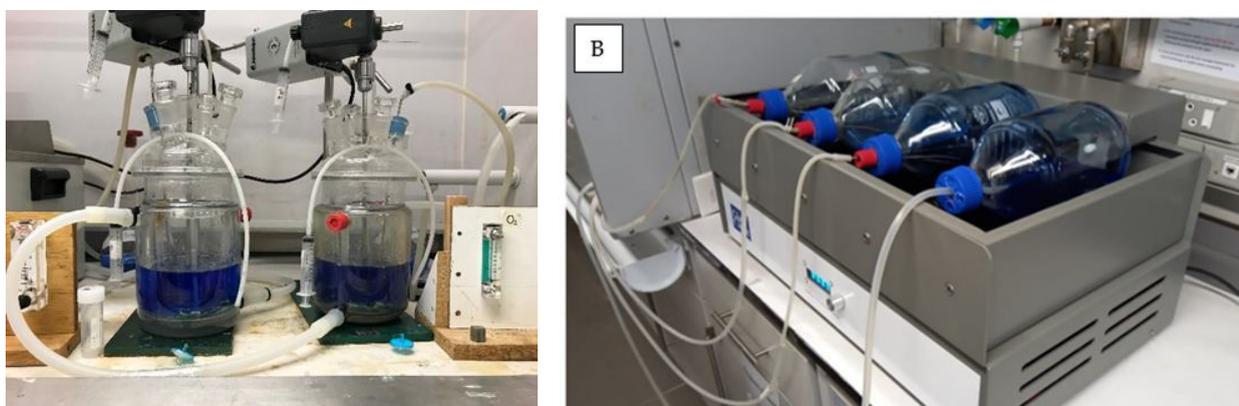


Figure 5. (A) Overhead-stirred glass reactors (B) Bottle roller experiments (with air supply)

3. RESULTS AND DISCUSSION

3.1. Evaluation of shredding cycles

The gradual comminution of an individual PCB with each shredding cycle is shown in Figure 6; the corresponding particle size distribution is given in Table 3

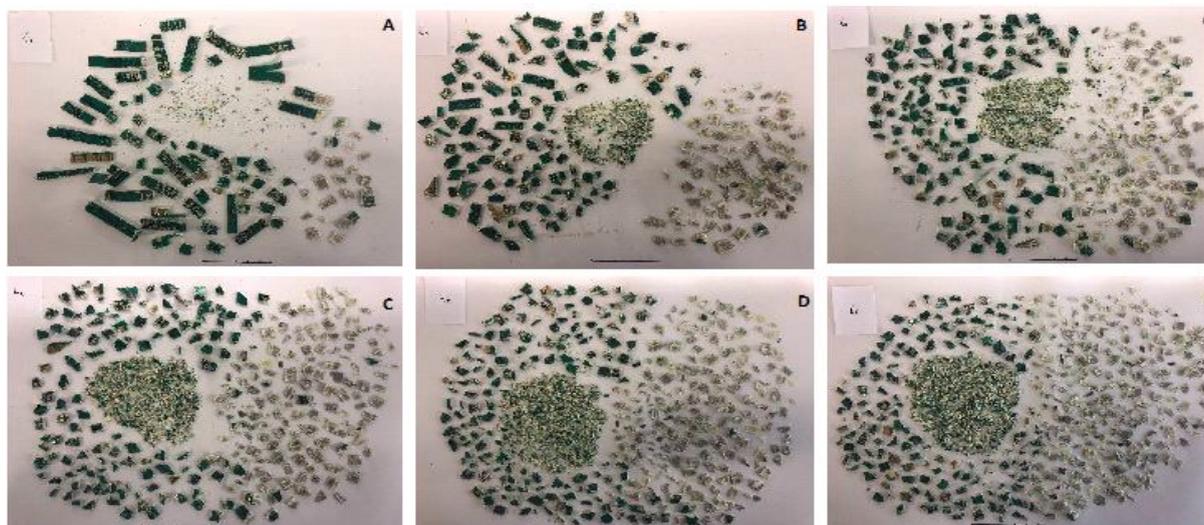


Figure 6. Pictures of all particles generated from consecutive shredding cycles (A: 1st shredding cycle to E: 6th shredding cycle)

Table 3. Particle size distribution after 1st to 6th shredding cycle

| Size class (µm) | 1 st | 2 nd | 3 rd | 4 th | 5 th | 6 th |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| >8000 | 86% | 63% | 49% | 44% | 32% | 26% |
| 6700 - 8000 | 4.6% | 11% | 14% | 16% | 19% | 21% |
| 4750 - 6700 | 6.5% | 13% | 22% | 20% | 25% | 25% |
| 3350 - 4750 | 1.3% | 5.9% | 6.5% | 9.5% | 11% | 13% |
| 2000 - 3350 | 0.78% | 3.0% | 3.7% | 5.3% | 6.6% | 7.0% |
| <2000 | 1.0% | 3.4% | 4.1% | 5.7% | 7.1% | 8.0% |

The result of measuring the power consumption over a total of 6 cycles is shown in Figure 7. The shredder operates at a base load of 118 W, which refers to energy losses in the motor and gear box. It can clearly be seen that when a board is fed through the shredder, the power consumption briefly increases until the board has been completely moved through the device. The power draw has been integrated over the length of each shredding cycle and the energy input, both per board and cycle, as well as per unit mass of board material, are given in Table 4. Cumulative energy input with increasing shredding cycle is also reflected.

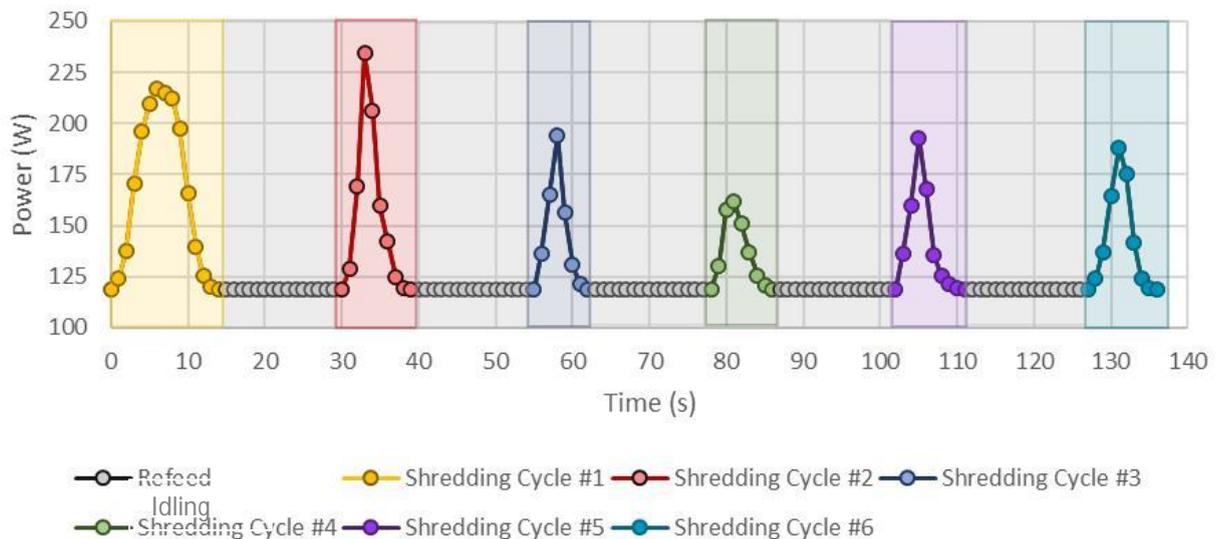
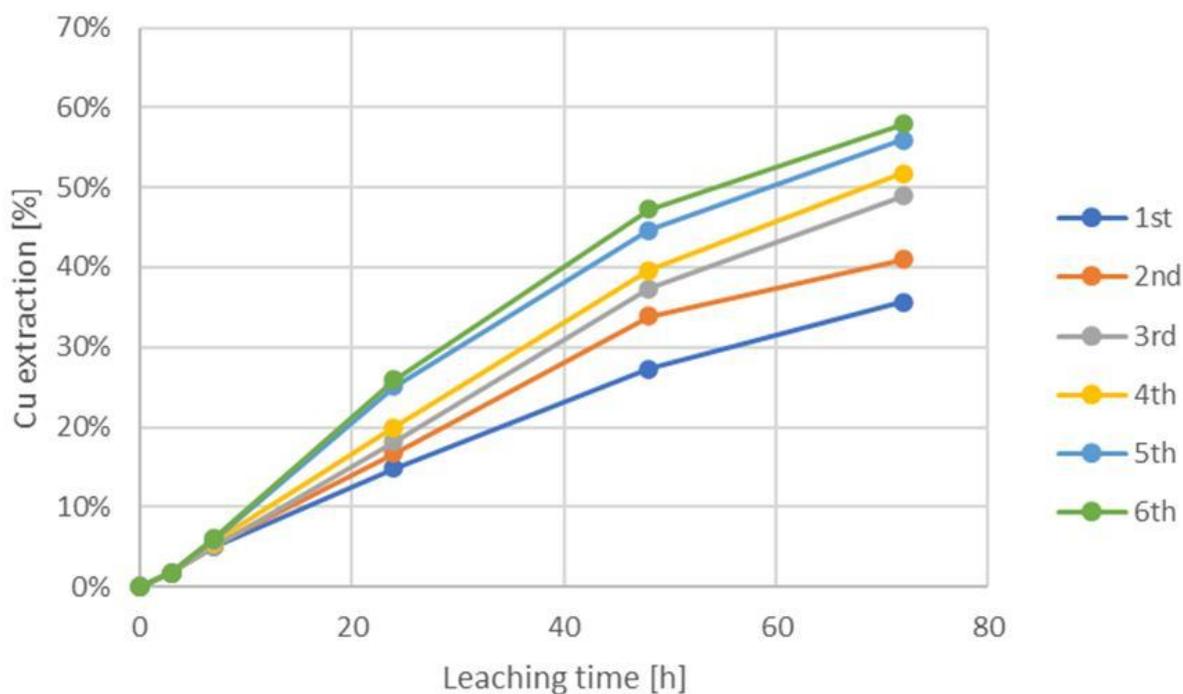


Figure 7. Power consumption of grab shredder during the 6 shredding cycles
 (NB: the idling power consumption between cycles indicates the base load of the device)

Table 4. Calculation of energy consumption per shredding cycle

| shredding cycle | cycle time (s) | Energy per board (J) | Energy per mass (kWh/kg) | Cumul. energy (kWh/kg) |
|-----------------|----------------|----------------------|--------------------------|------------------------|
| 1 st | 14 | 690 | 0.0037 | 0.0037 |
| 2 nd | 9 | 340 | 0.0018 | 0.0055 |
| 3 rd | 7 | 193 | 0.0010 | 0.0065 |
| 4 th | 7 | 158 | 0.0008 | 0.0073 |
| 5 th | 9 | 209 | 0.0011 | 0.0085 |
| 6 th | 9 | 237 | 0.0013 | 0.0097 |

The leach curves for the shredded samples using the areated stirred reactor set-up are shown in Figure 8. As can be seen material is increasingly liberated with each additional cycle, resulting in improved extraction. The relatively slow progression of leaching should be noted for all curves – this is assumed to be related to the relatively slow penetration of lixiviant to leach copper from between the different layers of the board. This is illustrated in Figure 9 which shows the unleached inner core layer of copper within a piece of shredded particle, which was peeled open.

**Figure 8. Cu extraction curves for the PCB material after different number of shredding cycles**

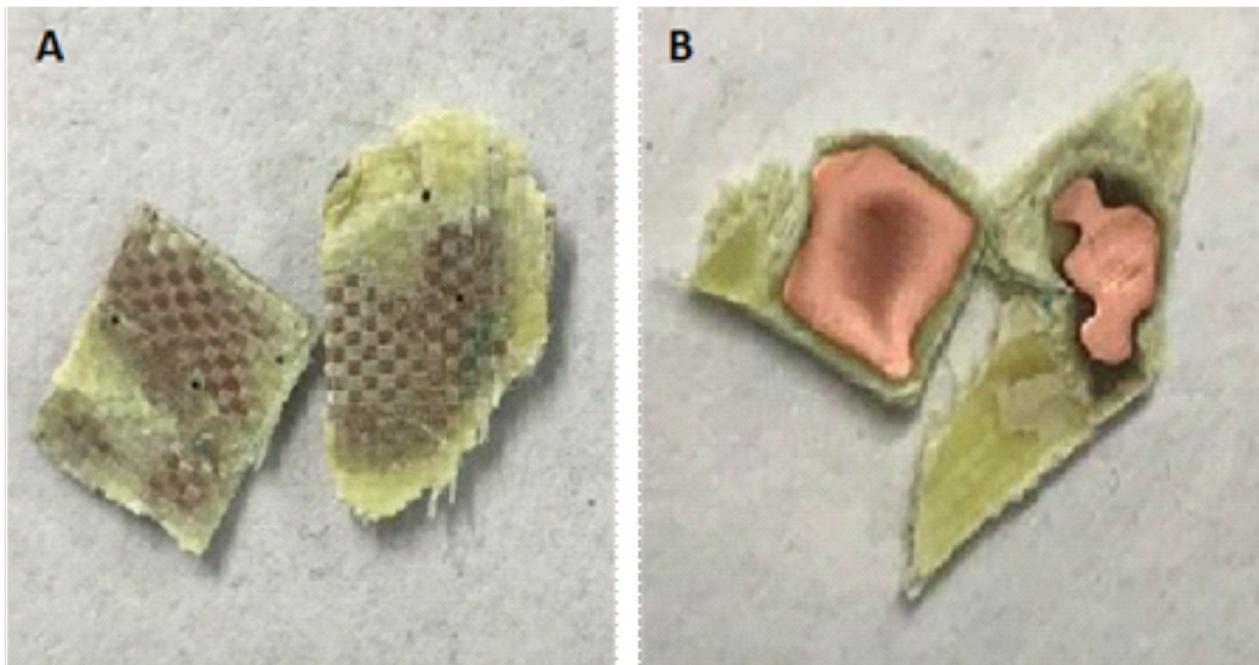


Figure 9. PCB particles (A) before leaching and (B) after leaching, peeled open

3.2. Evaluation of solvents

The results of the solvent screening tests are indicated in Table 5, showing that relative to untreated sample extraction was noticeably improved, with all reagents showing improvements, but NMP shows the best result (62% relative to 53% in the base case). NMP was therefore selected as the reagent for further exploration.

Table 5. Matrix of solvent screening tests

| Solvent | | Water | DMSO | NMP | 'E-Prep' |
|----------------|-----|-------|------|-----|----------|
| Temperature | °C | 90 | 90 | 90 | 90 |
| Residence Time | min | 75 | 75 | 75 | 75 |
| Cu Extraction | | 53% | 57% | 62% | 59% |

Table 6 shows the results of the tests in which the temperature for the impregnation reaction was varied and Table 7 those of where the impregnation time was varied. This work indicates that both increasing temperature and longer exposure to the swelling reagent improve the result, but the gains are not very significant. An operating value of 100°C and a residence time of 90 minutes was chosen as the operating conditions for the subsequent tests.

Table 6. Matrix of NMP extraction at various impregnation temperatures

| Organic Solvent | | NMP | NMP | NMP | NMP |
|-----------------|-----|-------|-------|-------|-------|
| Temperature | °C | 80 | 90 | 100 | 110 |
| Residence Time | min | 90 | 90 | 90 | 90 |
| Cu Extraction | | 54.4% | 53.3% | 55.6% | 61.6% |

Table 7. Matrix of NMP extraction at various impregnation residence times

| Organic Solvent | | NMP | NMP | NMP | NMP |
|-----------------|-----|-------|-------|-------|-------|
| Temperature | °C | 100 | 100 | 100 | 100 |
| Residence Time | min | 30 | 60 | 90 | 120 |
| Cu Extraction | | 55.0% | 55.1% | 55.6% | 62.3% |

3.3. Pre-treatment methods in comparison

Samples that were cut using a band saw had modest material losses at 5-6%, which were due to dust generated by the band saw. As these pieces did not undergo any other pre-treatment, there is no visible Cu. The Cu that becomes accessible for contact with the lixiviant sits under the gold plating which was found to flake off when the PCBs were put in the $(\text{NH}_4)_2\text{CO}_3$ - NH_4OH - CuSO_4 solutions. This exposes fractions of the top and bottom layers of Cu in the four-layer Cu sample used in the test. The PCB did not show any significant removal of the solder mask, which prevents metal-lixiviant contact and thus hinders leaching.

Shredded PCBs resulted in a sample of a wide particle size distribution, with the largest fraction of samples 44% reporting to $> 8000 \mu\text{m}$. The industrial grab shredder was expected to liberate copper through delamination and through size reduction. Effective delamination in this case refers to PCBs in which the layers in the board separated leaving at least one side of the PCB piece with exposed Cu. All particles in the image (Figure 10) were arranged such that any delaminated surfaces were facing up. Careful visual analysis of the PCBs showed that a significant quantity of particles remained intact while most of the delaminated particles were only partially delaminated with fibreglass still covering the metal surface. This is expected to hinder leaching owing to the impervious nature of the tightly laminated fibreglass and the set epoxy resin. The smaller particle sizes displayed liberated Cu.

A combination of cutting the boards and organic swelling (Method 3) showed the PCBs to have lost the outer layer of solder mask on surfaces on which the solder mask was in contact with metal (Figure 10.A). The solder mask remained intact on surfaces in which it had set before impregnation. Close inspection of the PCB pieces indicated the layers to show some extent of partial delamination of the layers even though the overall PCB piece remained intact (Figure 10.B).

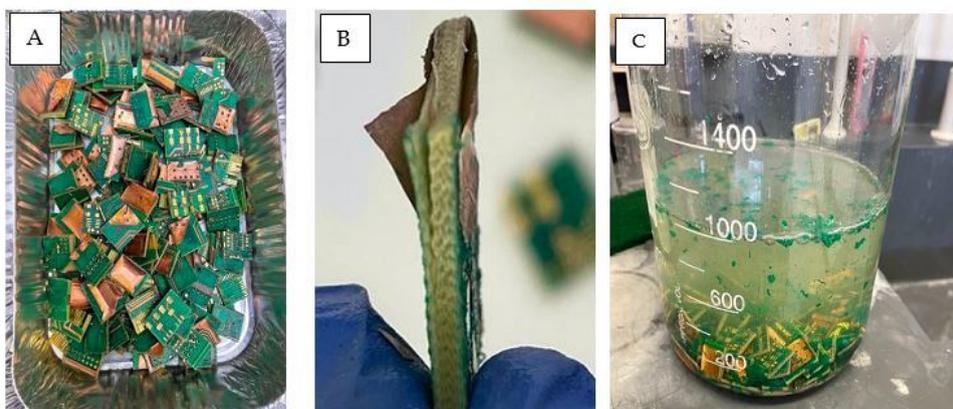


Figure 10. Cut PCB pieces after swelling in NMP at 100 °C for 90 min. (A) all the pieces from one PCB, (B) one PCB piece showing partial delamination, (C) PCB pieces in NMP showing solder mask floating.

PCBs that had passed through the shredder 4 times and undergone solvent swelling (Method 4) displayed similar attributes to the cut and organic solvent swelled PCBs, except that since these particles were relatively smaller and some of them already partially delaminated, the delamination appeared more effective.

Leach tests were run as described in the methodology section. The results (see Figure 11) show that when the board are cut (Method 1) the copper extraction reached only 15%, which is low, since the leaching solution could not reach the copper hidden between the layers of the boards (see Figure 3). However, when the boards were cut and then swelled in the organic reagent (Method 3), the leaching kinetics improved, achieving 44 % of extraction, largely due to the removal of the solder mask, floating on top of the swelling reagent solution (see Figure 10.C), exposing copper in to top and bottom layer, but also due to copper intermediate layers becoming more exposed to the leaching solution as a result of the swelling/delamination as can be seen in Figure 10.B.

When the boards were shredded (Method 2) achieving a copper extraction of 51% thanks to the size reduction observed in figure 6. Finally, the boards that were shredded and then swelled in the organic reagent achieved an extraction of 68%, indicating that combination of the two methods yields the best effect. However, in all curves it appears the leaching is delayed for the first 3 hours, which is a strong indication that the initial 100 ppm copper was still too little to facilitate the catalytic role for copper oxidation.

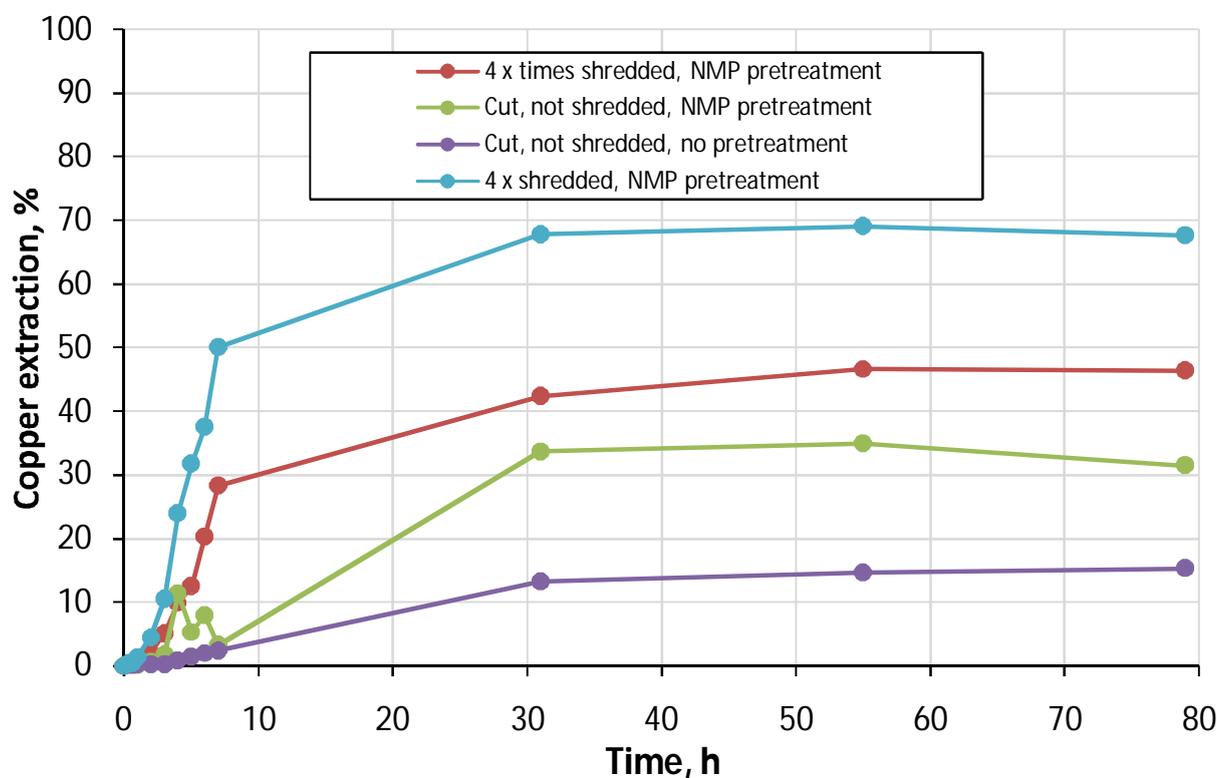


Figure 11. Results of the comparative leaching experiments

4. CONCLUSIONS

In the present study we presented a series of experiments around the preparation of model PCBs for Cu leaching in an ammonia system. While most studies finely grind the PCB material to a fine powder prior to leaching, this approach is also by far the most energy intensive step, prompting this research to identify milder routes. Shredding the PCBs in combination with swelling the cut boards in NMP, an organic reactant that can easily be recovered through distillation of the spent solution, has shown to yield close to 70% of the contained copper. As was shown here, shredding of the boards is quite effective and has very modest energy consumption. Increasing solvent soaking time and temperature also improve the extraction, but shredding prior to impregnation is an equally critical fact as in combination excellent extraction results are achievable.

It is noted, however, that process starts slow and could potentially be enhanced at much higher starting concentrations as is industrial practice. All leach tests were also conducted at ambient temperatures and hot (>50°C) leaching has as yet not been explored for this system and should be subject for further investigation.

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