

Analysis

EC Dismantling

Pickling Solution

Table 1 summarizes the results of different concentrations of nitric acid in the pickling solution on EC removal. Only the HNO₃ concentration was varied. The time needed to fully remove ECs and the weight of PCBs before and after EC removal were included in Table 1. Figure 4 shows the experiment setup.

Table 1

Impact of Nitric Acid Concentration on EC Removal

PCB starting weight (g)	HNO ₃ concentration (vol%)	Time (minutes)	PCB end weight (g)
1.09	40	38	0.93
0.739	35	70	0.60
0.839	30	Did not fully remove EC after 90 minutes	N/A
0.839	25	Did not fully remove EC after 90 minutes	N/A
0.709	20	Did not fully remove EC after 90 minutes	N/A

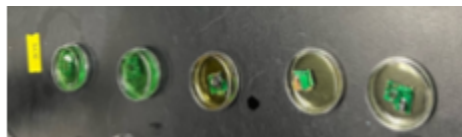


Figure 4: The impact of nitric acid concentration on EC removal.

From left to right: 40%, 35%, 30%, 25% and 20%.

Table 2

EC Removal by Pickling Solution with 35 vol% Nitric Acid at Large Scale

Round No.	Fresh PCBs before reaction (g)	Reaction time (min)	PCBs after reaction (g)	ECs collected(g)
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1	127	72	97	22
2	126	68	85	31
3	127	66	94	28

Table 3 summarizes the time recorded to fully remove ECs from PCBs in both small scale and large-scale experiments. Table 4 shows the correlation analysis results of data in Table 3.

Table 3

Time to Remove ECs by Pickling Solutions with different Nitric Acid Concentrations

HNO ₃ concentration (vol%)	Time (minutes)
40	38
35	70
35	72
35	68
35	66
35	60

Table 4

Correlation Analysis of Data in Table 3

Correlations			
		HNO ₃ concentration (vol%)	Time (minutes)
HNO ₃ concentration (vol%)	Pearson Correlation	1	-.945**
	Sig. (2-tailed)		.004
	N	6	6
Time (minutes)	Pearson Correlation	-.945**	1
	Sig. (2-tailed)	.004	
	N	6	6

** . Correlation is significant at the 0.01 level (2-tailed).

Fluoroboric Acid with Hydrogen Peroxide

Table 5 summarizes the effectiveness of different S/L ratios for dismantling ECs by the HBF_4 + H_2O_2 recipe. The S/L ratios ranged from 1g:2ml to 1g:9ml.

Table 5

Effect of S/L Ratio on EC Removal for HBF_4 + H_2O_2 Recipe

test number	S/L ratio (g:ml)	EC removed?
1	1:2	N
2	1:3	N
3	1:4	N
4	1:5	Y
5	1:6	Y
6	1:7.5	Y
7	1:9	Y

Solder Mask Removal***NaOH vs. KOH***

Figure 5 compares the results of PWBs treated with 2M NaOH and 2M KOH to remove the green solder masks on their surface.

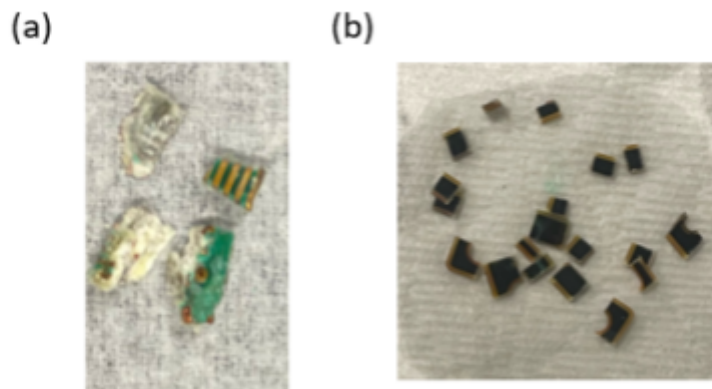


Figure 5: PWBs treated (a) PWBs with 2M NaOH at 90°C for 90 min. (b) with 2M KOH at 90°C for 90 min.

Optimizing KOH Recipe

Figure 6 shows the experiment setup to optimize KOH concentration and S/L ratio for solder mask removal. Figures 7 and 8 compare the results of KOH concentration and S/L ratio tests.



Figure 6: Tests to compare 1M and 2M KOH at different S/L ratios. Group 1 (top row) is 1M KOH. Group 2 (bottom row) is 2M KOH. From left to right are S/L ratios: 1g:1ml, 1g:2ml, 1g:3ml, 1g:4ml, 1g:5ml, respectively.



Figure 7: Results of 1M KOH. From left to right S/L ratios (g:ml): 1:1, 1:2, 1:3, 1:4, 1:5, respectively.

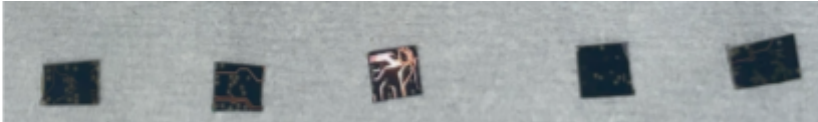


Figure 8: Results of 2M KOH. From left to right S/L ratios (g:ml): 1:1, 1:2, 1:3, 1:4, 1:5, respectively.

Mechanical Shredding

Figure 9 shows the shredded clean PWBs without solder masks.

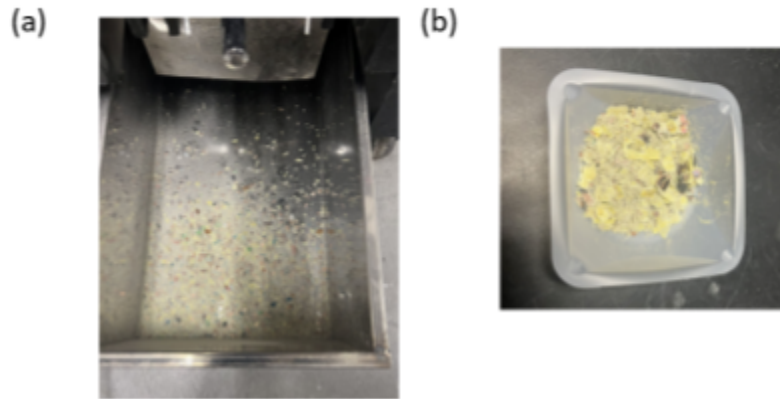


Figure 9: (a) Shredded PWBs in the bottom collecting container of the mechanical shredder. (b) Shredded PWBs close-up. Weight: 2g.

Dense Medium Separation

Figure 10 compares the dense medium separation experiment results with TBE to acetone volume ratio ranging from 10ml:2ml to 10ml:10ml.

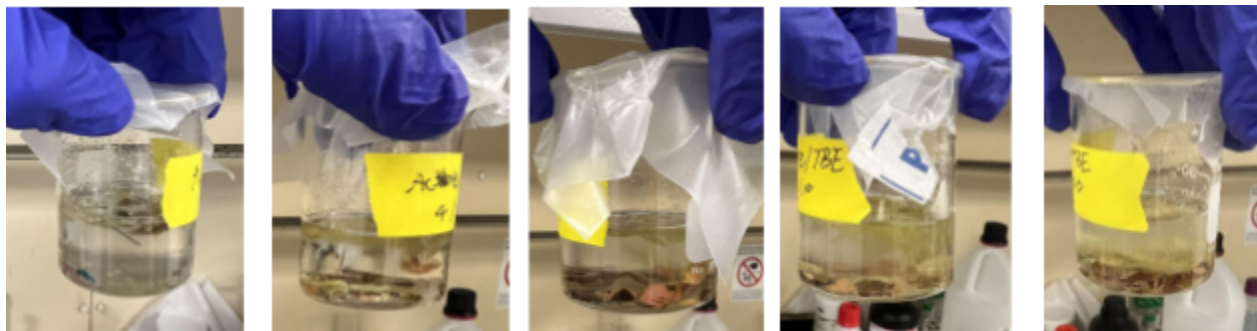


Figure 10: Separation experiments using TBE to acetone volume ratios (ml:ml): 10:2, 10:4, 10:6, 10:8, 10:10, from left to right, respectively.

Reuse of Chemicals

Reuse of Pickling Solutions

Table 6 summarizes the results of the experiments to test the effectiveness of reused pickling solutions in removing ECs. The weights of PCBs before and after pickling solution treatment, the weight of ECs collected, and the time needed to fully remove ECs were included in Table 6.

Table 6

Summary of the Effectiveness of Reused Pickling Solutions

Round No.	Weight of PCBs before reaction (g)	Time to remove ECs	Weight of PCBs after reaction (g)	Weight of ECs collected (g)
1	3.4429	60 mins	1.5608	1.6294
2	2.7826	75 mins	2.0458	0.5434
3	3.2563	Left overnight and removed after overnight	2.5032	0.5379

Reuse of KOH

Table 7 summarizes the results of the experiments to test the effectiveness of reused KOH on removing solder masks. The weights of PWBs before and after KOH treatment, the weight of solder masks removed, the volume of KOH after filtration in each round, and the time needed to fully remove solder masks were included in Table 4.

Table 7

Summary of the effectiveness of reused KOH solutions

Round No.	PWBs before reaction	Reaction time	PWBs after reaction	Solder mask removed	KOH after filtration	Fresh KOH added
1	138 g	90 mins	132 g	6 g	250 ml	50 ml
2	164 g	90 mins	159 g	5 g	250 ml	45 ml

3	152 g	90 mins	147 g	5 g	260 ml	40 ml
4	152 g	90 mins	146 g	6 g	255 ml	N/A

Reuse of TBE/Acetone

Table 8 summarizes the results of the experiments to test the effectiveness of reused TBE/Acetone solutions when separating the shredded pieces. The table includes each round’s weights of shredded pieces used, the volume of solution loss after filtration, the volume of fresh TBE and acetone added, and the total volume of used solution.

Table 8

Summary of the Effectiveness of Reused TBE/Acetone Solutions

Round No.	Fresh TBM added (mL)	Fresh acetone added (mL)	Total solution (mL)	Weight of shredded pieces (g)	Solution loss after filtration (mL)	Separation effective?
1			18	2.1593	3.3	Y
2	1	1.7	17.4	2.1774	2.1	Y
3	1	1.7	18	2.3522	2.7	Y
4	1	1.7	18	2.2642	2.5	Y
5	1	1.7	18	2.2448	2.9	Y
6	1	1.7	18	2.1593	3.3	Y
7	1	1.7	17.4	2.1774	2.1	Y
8	1	1.7	18	2.3522	2.7	Y
9	1	1.7	18	2.2642	2.5	Y
10	1	1.7	17.2	1.8895	2.9	Y

Characterizations

EDX Result

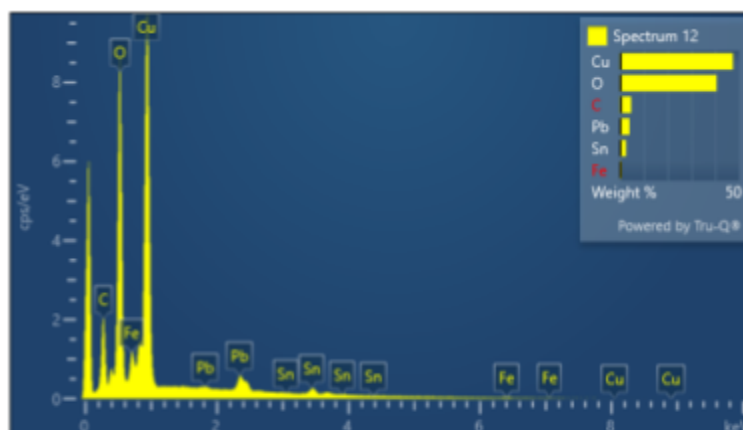


Figure 11 shows the EDX result of the precipitates from the EC dismantling by pickling solution process. The spectrum includes the peaks signaling different elements and their relative quantity.

ICP-OES Results

Table 9

Concentrations of Metals in Pickling Solutions After Different Steps of Treatment

	Cu (g/L)	Sn (g/L)	Fe (g/L)	Ni (g/L)	Pb (g/L)	Zn (g/L)
Fresh pickling solution	0	0	7.2	0	0	0
After 1st round of EC removal	10.6	0.56	8.6	0.65	0	0
After 3rd round of EC removal	38.8	3	14.1	2.5	2.9	2.9
After adding NaOH solution	0	0	0	0	0.02	0.02

Table 9 shows the concentration of different metals in pickling solutions after different steps of treatment, including in fresh pickling solution, after 1st round of EC removal experiment, after the 3rd round of EC removal experiment, and after adding NaOH to precipitate the metals post the 3rd round of EC removal. The concentrations were obtained by ICP-OES.