Electronic Supplementary Information (ESI)

Recovery of copper, zinc and lead from photovoltaic panel residue

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Figure S1: Photovoltaic panel residue (starting material)

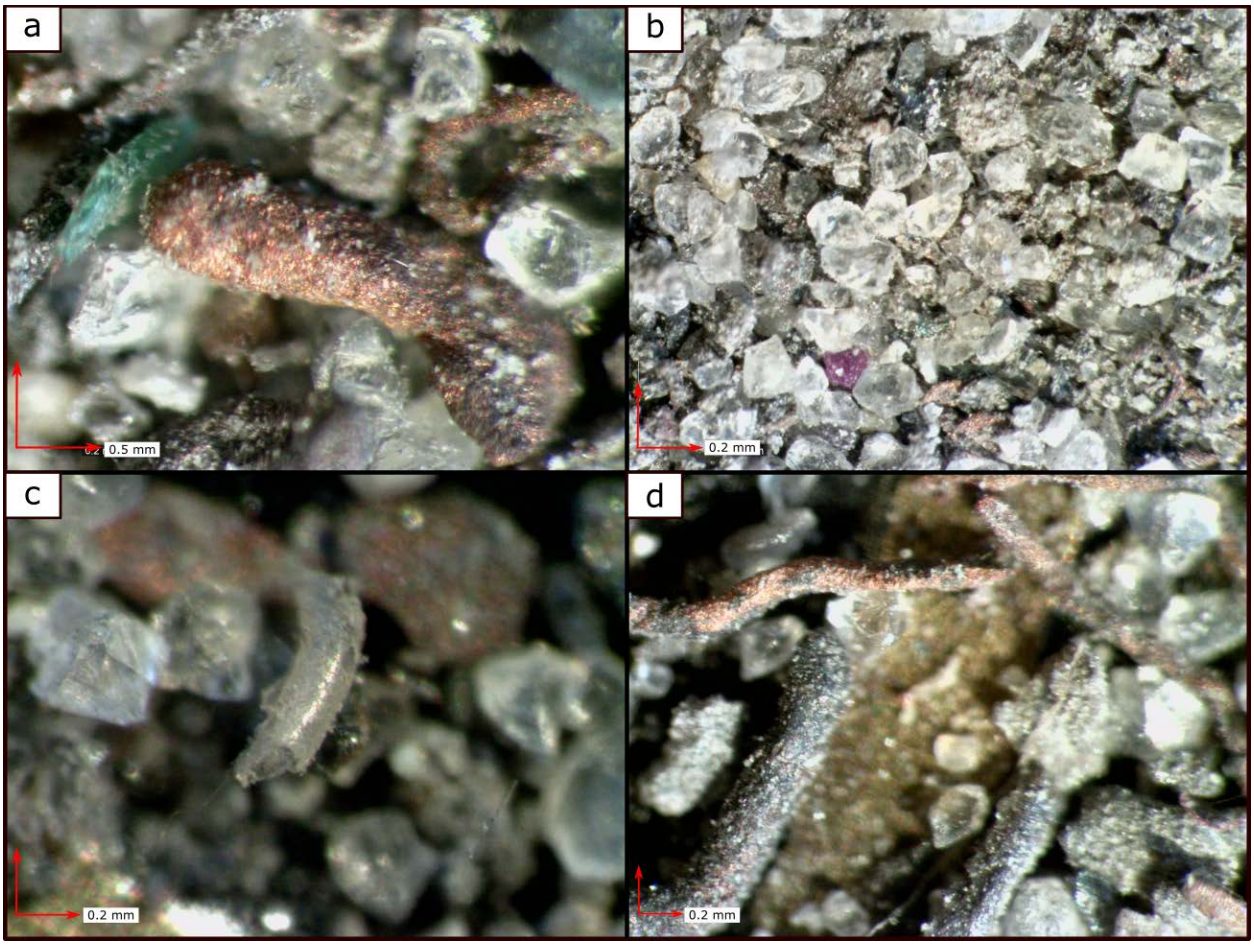

Figure S2: Stereo microscopic pictures of the starting material. (a) Cu wires and malachite, (b) PbO phase (red) and other Pb-bearing phases, (c) silicon tubes, (d) Cu wires and other alloy wires

Table S1. Copper, zinc and lead leaching efficiencies for 4^3 Box-Behnken design

Standard Order	Experimental Order	Independent Variables				Response [E_L (%)]				
		HCl (mol L^{-1})	NaCl (g L^{-1})	H_2O_2 (%wt)	time (min)	Cu	Zn	Pb	Fe	Ag
23	1	1.5	0	5	60	49.26	96.55	8.00	33.46	0.00
5	2	1.5	100	0	10	0.06	3.65	16.92	18.19	0.00
17	3	0	100	0	35	0.01	0.46	3.05	28.19	0.00
21	4	1.5	0	5	10	46.53	95.47	7.91	31.73	0.00
12	5	3	100	5	60	86.03	99.07	36.56	41.17	55.75
25	6	1.5	100	5	35	67.61	99.64	24.10	40.43	24.60
22	7	1.5	200	5	10	83.12	99.83	67.81	36.15	53.28
3	8	0	200	5	35	79.44	89.51	81.93	41.24	34.72
19	9	0	100	10	35	76.51	91.69	24.02	33.97	29.28
14	10	1.5	200	0	35	0.00	0.87	2.47	0.00	0.00
7	11	1.5	100	0	60	0.11	2.35	16.39	32.00	0.00
11	12	0	100	5	60	31.84	61.51	51.93	23.24	17.72
2	13	3	0	5	35	78.37	99.03	14.20	40.87	56.30
6	14	1.5	100	10	10	74.67	99.62	25.60	35.16	25.32
13	15	1.5	0	0	35	0.06	1.05	5.43	15.91	0.00
26	16	1.5	100	5	35	67.61	99.64	24.10	40.43	24.60
8	17	1.5	100	10	60	75.34	99.42	25.18	38.53	19.85
27	18	1.5	100	5	35	67.61	99.64	24.10	40.43	24.60
20	19	3	100	10	35	74.87	93.03	11.20	36.87	47.30
4	20	3	200	5	35	88.85	98.58	41.40	45.59	53.40
18	21	3	100	0	35	0.00	0.90	2.47	0.00	0.00
15	22	1.5	0	10	35	55.08	99.59	8.21	29.18	0.00
10	23	3	100	5	10	86.05	95.58	50.40	40.59	40.40
1	24	0	0	5	35	0.00	0.83	2.47	0.00	0.00
16	25	1.5	200	10	35	57.81	70.64	41.10	35.43	12.60
9	26	0	100	5	10	18.54	44.51	27.93	10.24	12.72
24	27	1.5	200	5	60	80.54	96.80	69.41	36.48	47.24

Response Surface Regression: Cu versus A, B, C, D

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	14	26478.6	1891.3	6.09	0.002
Linear	4	20048.7	5012.2	16.14	0.000
A	1	3600.0	3600.0	11.59	0.005
B	1	2145.7	2145.7	6.91	0.022
C	1	14286.2	14286.2	45.99	0.000
D	1	16.7	16.7	0.05	0.821
Square	4	5187.0	1296.8	4.17	0.024
A*A	1	79.9	79.9	0.26	0.621
B*B	1	87.5	87.5	0.28	0.605
C*C	1	4679.1	4679.1	15.06	0.002
D*D	1	31.4	31.4	0.10	0.756
2-Way Interaction	6	1243.0	207.2	0.67	0.678
A*B	1	1188.8	1188.8	3.83	0.074
A*C	1	0.7	0.7	0.00	0.964
A*D	1	44.4	44.4	0.14	0.712
B*C	1	1.9	1.9	0.01	0.938
B*D	1	7.0	7.0	0.02	0.883
C*D	1	0.1	0.1	0.00	0.986
Error	12	3727.6	310.6		
Lack-of-Fit	10	3727.6	372.8	*	*
Pure Error	2	0.0	0.0		
Total	26	30206.2			

Model Summary

S	R-sq
17.6247	87.66%

$$Cu = -64.8 + 31.6\,A + 0.399\,B + 18.65\,C + 0.50\,D - 1.72\,A^*A - 0.000405\,B^*B - 1.185\,C^*C - 0.0039\,D^*D - 0.1149\,A^*B - 0.05\,A^*C - 0.089\,A^*D + 0.0014\,B^*C - 0.00053\,B^*D + 0.0012\,C^*D$$

Figure S3: Response surface regression analysis for Cu leaching efficiency; A = HCl, B = NaCl, C = H_2O_2 and D = time. (Minitab version 17)

Response Surface Regression: Zn versus A, B, C, D

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	14	43170.4	3083.6	8.46	0.000
Linear	4	29499.8	7375.0	20.23	0.000
A	1	3423.5	3423.5	9.39	0.010
B	1	317.3	317.3	0.87	0.369
C	1	25734.8	25734.8	70.58	0.000
D	1	24.2	24.2	0.07	0.801
Square	4	11389.1	2847.3	7.81	0.002
A*A	1	1673.4	1673.4	4.59	0.053
B*B	1	416.0	416.0	1.14	0.307
C*C	1	10041.9	10041.9	27.54	0.000
D*D	1	14.2	14.2	0.04	0.847
2-Way Interaction	6	2281.4	380.2	1.04	0.445
A*B	1	2030.8	2030.8	5.57	0.036
A*C	1	0.2	0.2	0.00	0.981
A*D	1	22.6	22.6	0.06	0.808
B*C	1	221.5	221.5	0.61	0.451
B*D	1	4.2	4.2	0.01	0.916
C*D	1	2.1	2.1	0.01	0.941
Error	12	4375.7	364.6		
Lack-of-Fit	10	4375.7	437.6	*	*
Pure Error	2	0.0	0.0		
Total	26	47546.1			

Model Summary

S	R-sq
19.0956	90.80%

Regression Equation in Uncoded Units

$$Zn = -78.4 + 52.0\,A + 0.542\,B + 28.26\,C + 0.40\,D - 7.87\,A^*A - 0.000883\,B^*B - 1.736\,C^*C - 0.0026\,D^*D - 0.1502\,A^*B + 0.03\,A^*C - 0.063\,A^*D - 0.0149\,B^*C - 0.00041\,B^*D - 0.0058\,C^*D$$

Figure S4: Response surface regression analysis for Zn leaching efficiency; A = HCl, B = NaCl, C = H_2O_2 and D = time. (Minitab version 17)

Response Surface Regression: Pb versus A, B, C, D

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	14	9989.8	713.55	4.11	0.009
Linear	4	5336.8	1334.20	7.68	0.003
A	1	184.7	184.70	1.06	0.323
B	1	4716.5	4716.53	27.14	0.000
C	1	391.9	391.86	2.26	0.159
D	1	43.7	43.71	0.25	0.625
Square	4	3702.7	925.68	5.33	0.011
A*A	1	107.1	107.09	0.62	0.448
B*B	1	78.1	78.10	0.45	0.515
C*C	1	1579.4	1579.36	9.09	0.011
D*D	1	710.1	710.11	4.09	0.066
2-Way Interaction	6	950.2	158.37	0.91	0.519
A*B	1	682.6	682.57	3.93	0.071
A*C	1	37.5	37.45	0.22	0.651
A*D	1	166.9	166.95	0.96	0.346
B*C	1	62.7	62.71	0.36	0.559
B*D	1	0.6	0.57	0.00	0.955
C*D	1	0.0	0.00	0.00	0.997
Error	12	2085.3	173.77		
Lack-of-Fit	10	2085.3	208.53	*	*
Pure Error	2	0.0	0.00		
Total	26	12075.0			

Model Summary

S	R-sq
13.1823	82.73%

Regression Equation in Uncoded Units

Pb = -7.1 + 8.2 A + 0.207 B + 7.84 C - 0.974 D + 1.99 A*A + 0.000383 B*B - 0.688 C*C
+ 0.01846 D*D - 0.0871 A*B - 0.408 A*C - 0.172 A*D + 0.0079 B*C + 0.00015 B*D
+ 0.0002 C*D

Figure S5: Response surface regression analysis for Pb leaching efficiency; A = HCl, B = NaCl, C = H_2O_2 and D = time. (Minitab version 17)

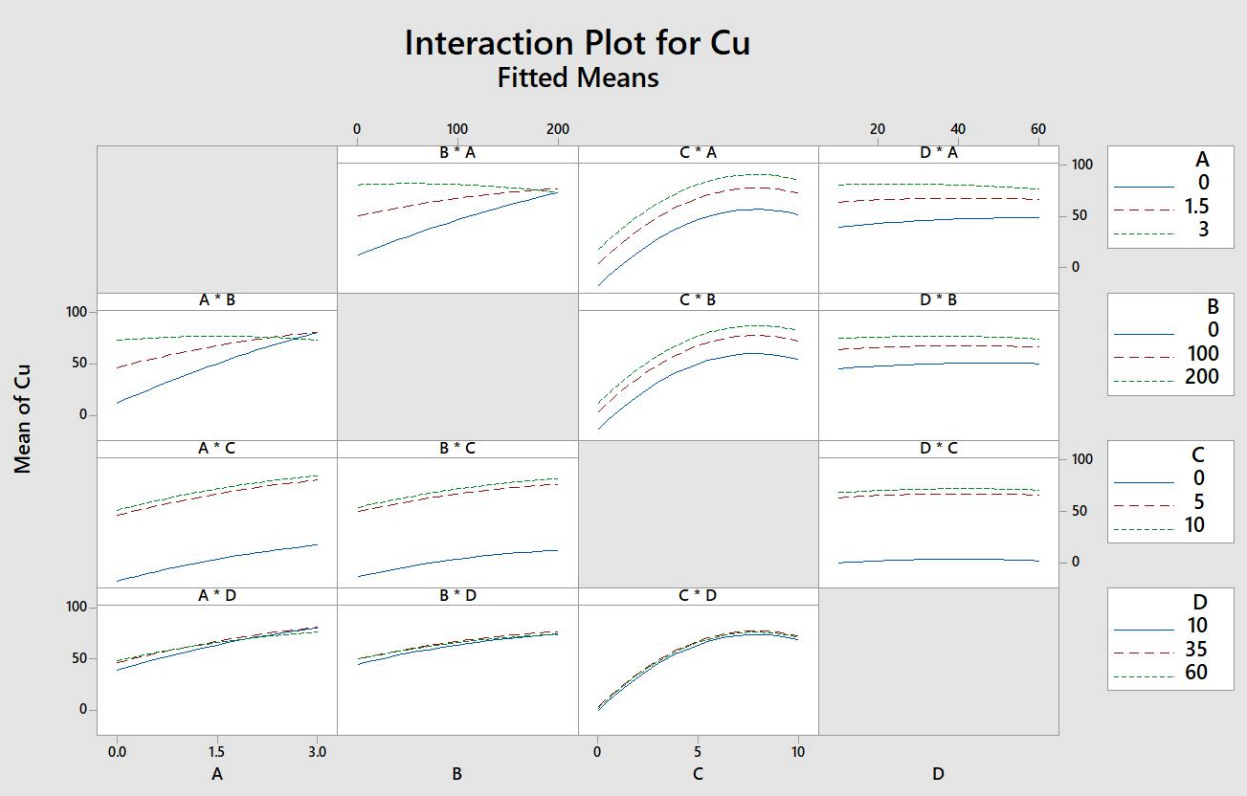

Figure S6: Interaction plot for Cu leaching efficiency. A = HCl, B = NaCl, C = H_2O_2 and D = time. (Minitab version 17)

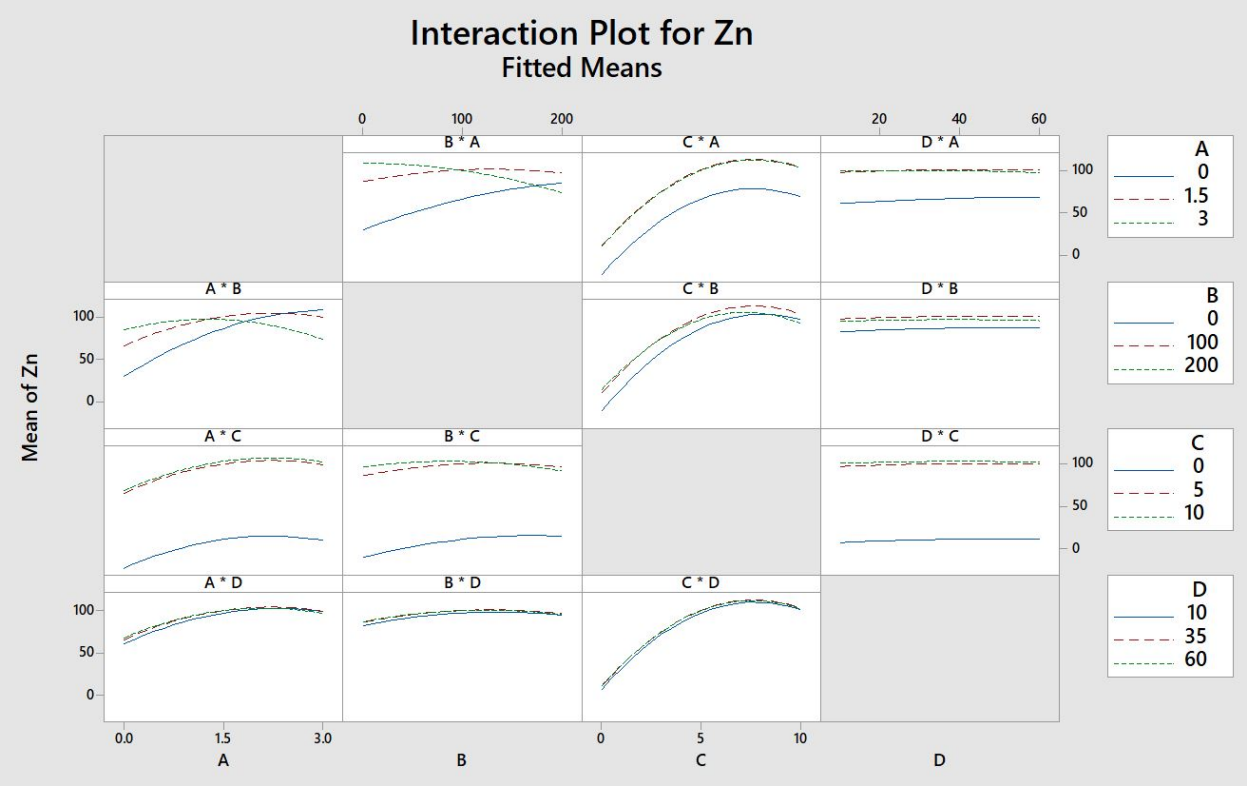

Figure S7: Interaction plot for Zn leaching efficiency. A = HCl, B = NaCl, C = H_2O_2 and D = time. (Minitab version 17)

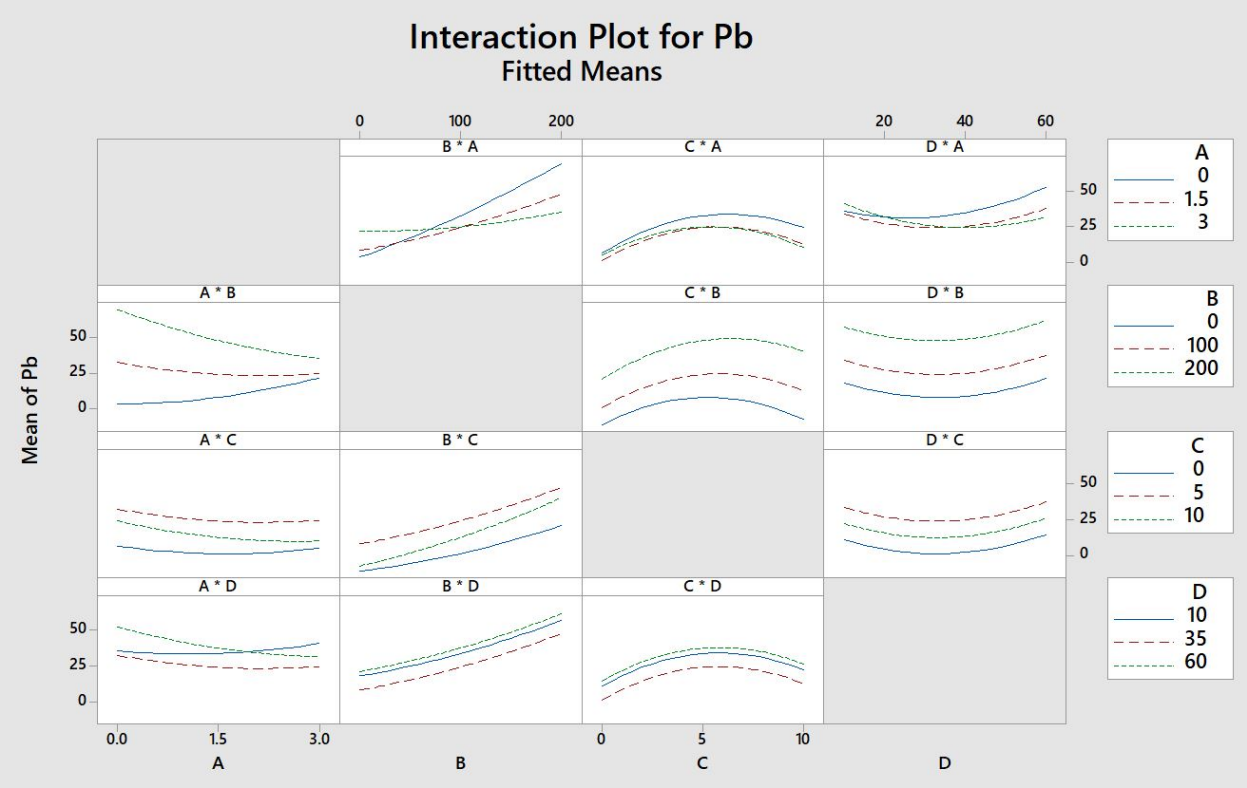

Figure S8: Interaction plot for Pb leaching efficiency. A = HCl, B = NaCl, C = H_2O_2 and D = time. (Minitab version 17)

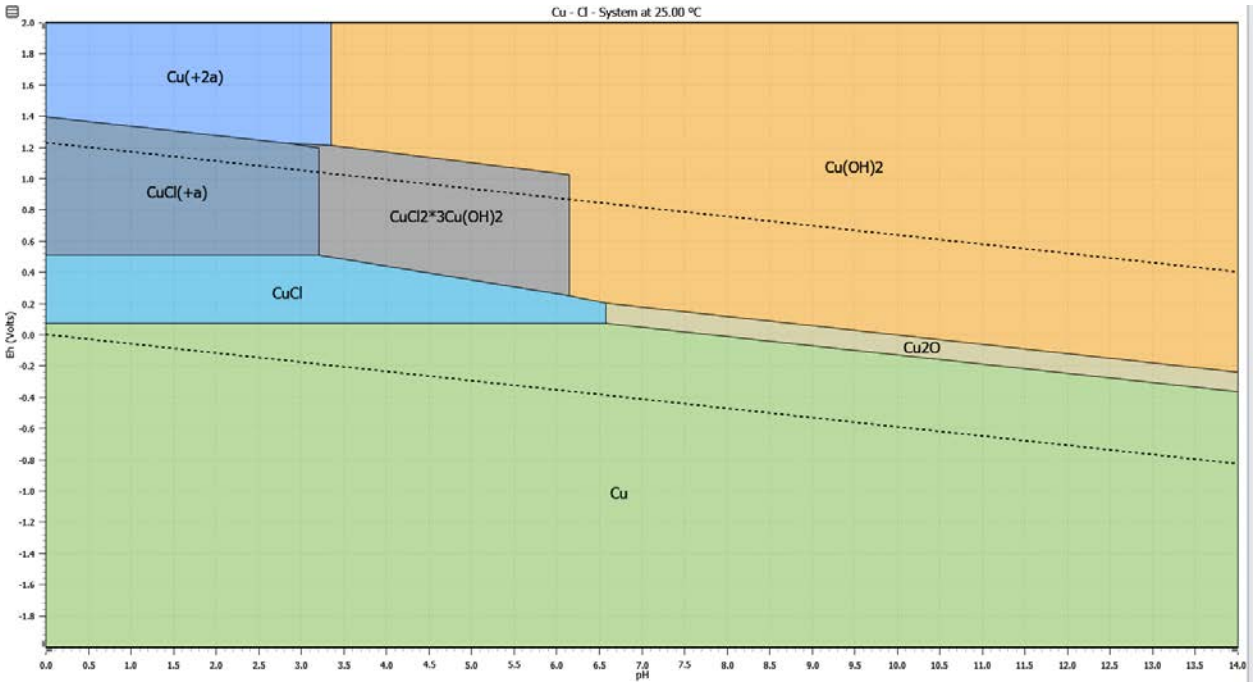

Figure S9: Eh-pH diagram for Cu (0.5 mol L^{-1}) in 1.5 mol L^{-1} HCl and 200 g L^{-1} NaCl at 25 °C (HSC Chemistry version 10). The Cu concentration was determined by ICP-OES analyses of the leachates.

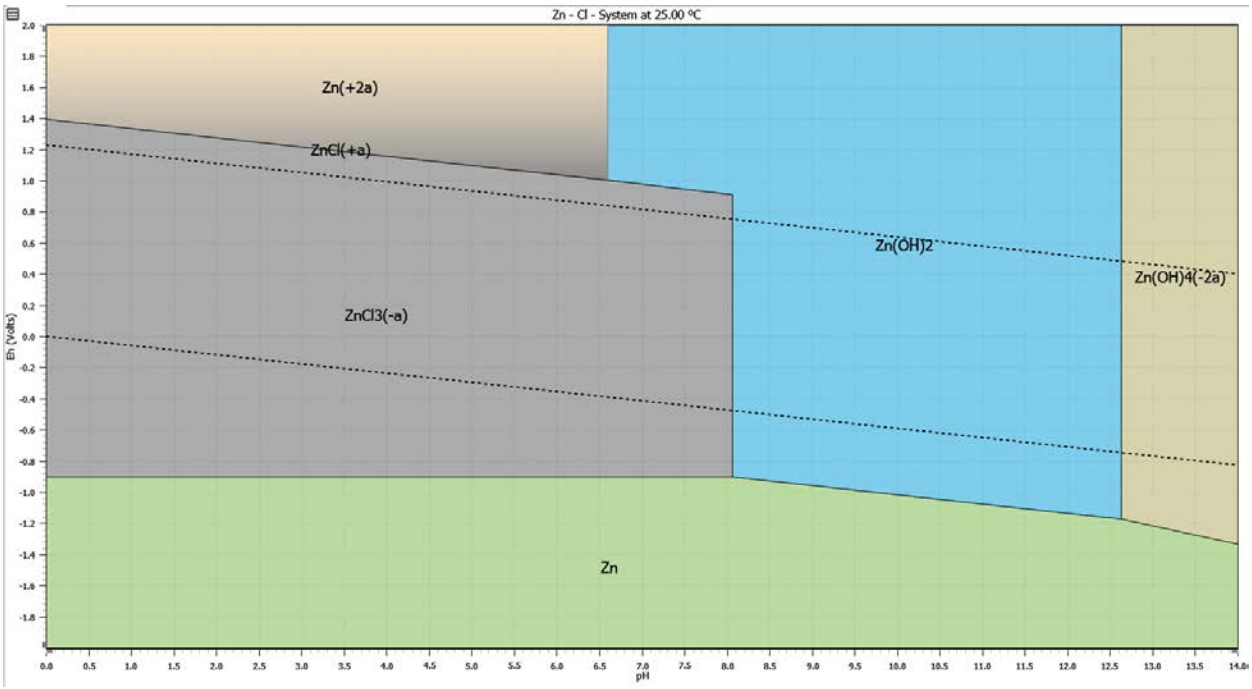

Figure S10: Eh-pH diagram for Zn (0.02 mol L^{-1}) in 1.5 mol L^{-1} HCL and 200 g L^{-1} NaCl at 25 °C (HSC Chemistry version 10). The Zn concentration was determined by ICP-OES analyses of the leachates.

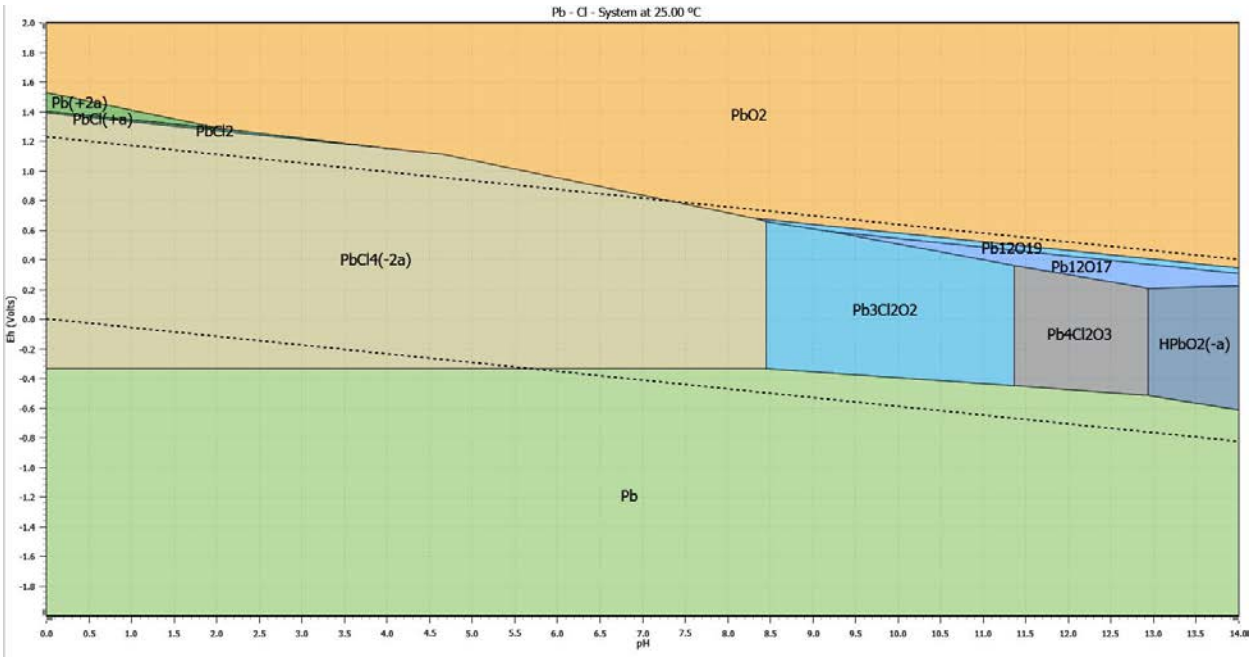

Figure S11: Eh-pH diagram for Pb (0.004 mol L^{-1}) in 1.5 mol L^{-1} HCl and 200 g L^{-1} NaCl at 25 °C (HSC Chemistry version 10). The Pb concentration was determined by ICP-OES analyses of the leachates.

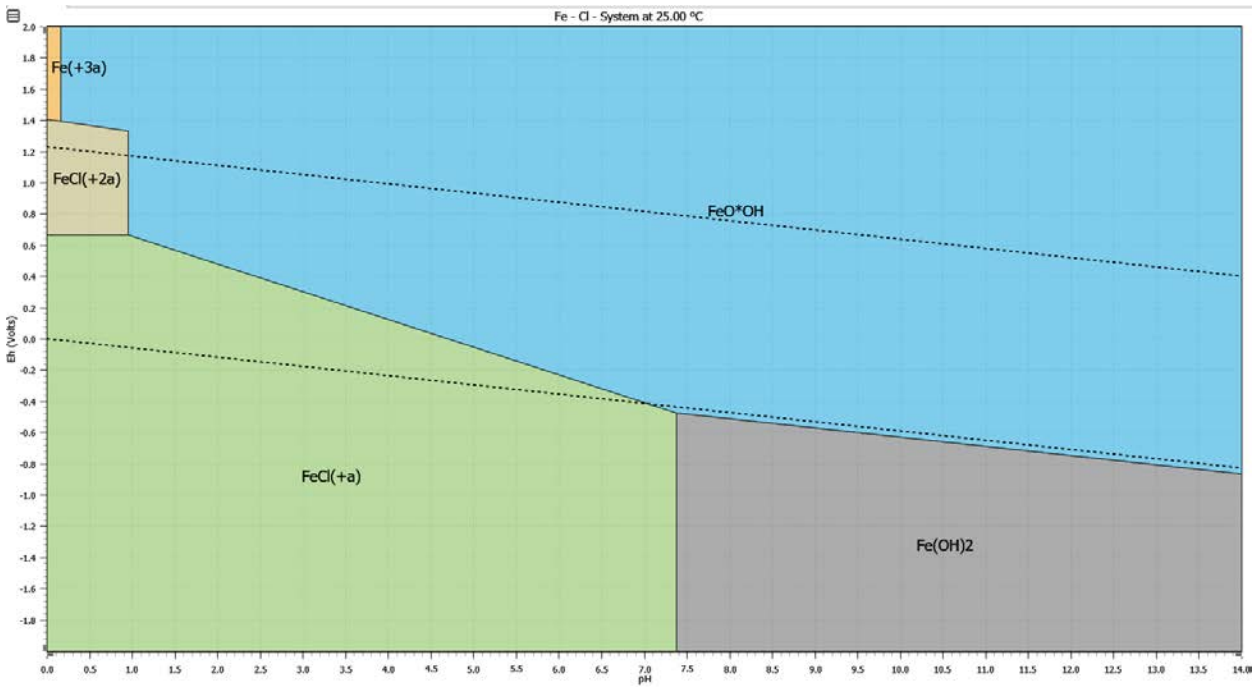

Figure S12: Eh-pH diagram for Fe (0.5 mol L^{-1}) in 1.5 mol L^{-1} HCl and 200 g L^{-1} NaCl at 25 °C (HSC Chemistry version 10). The Fe concentration was determined by ICP-OES analyses of the solution after the cementation step.

Table S2: Main elemental composition of microwave-assisted leaching solid residue (leaching conditions: [HCl] = 0.5 mol L^{-1}, [NaCl] = 200 g L^{-1}, [H_2O_2] = 7.5 wt% and t = 60 min)

Element	**wt%**	**Element**	**wt%**
Ag	0.1	Na	3.2
Al	0.8	Ni	0.6
Ba	0.6	Pb	6.2
Ca	0.9	Sb	0.2
Cr	0.7	Si	4.5
Cu	9.2	Sn	0.8
Fe	3.1	Ti	0.1
Mg	0.2	Zn	n.d.
Mn	0.2		

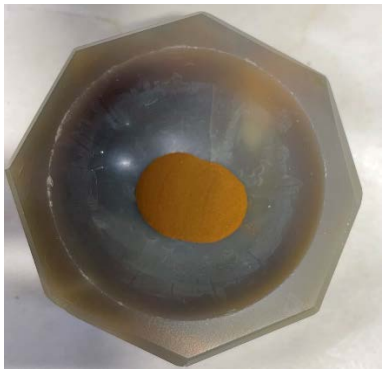

Figure S13: Fe-Cu cementation product

Figure S14: Pb sublate

Figure S15: Fe-rich precipitate

Figure S16: Zn-rich precipitate

Table S3: Chemical composition of the solution after Zn precipitation

Element	**mg L^{-1}**	**Element**	**mg L^{-1}**
Ag	n.d	Na	528±15
Al	113±7	Ni	<1
Ba	<1	Pb	n.d
Ca	28±3	Sb	7±1
Cr	n.d	Si	<1
Cu	n.d	Sn	12±2
Fe	<1	Ti	<1
Mg	37±4	Zn	n.d.
Mn	<1		