

Supplementary Data for *Journal of Sustainable Metallurgy*

Translating Selective Lithium Leaching from LFP to Industrial Black

Mass: Experimental Validation and Statistical Quantification

Daniel Reyes-Martinez · Ilias Lampropoulos · Rafaella Aikaterini Megaloudi · Alexandra Thiere ·

Lesia Sandig-Predzymirska · Panagiotis Xanthopoulos · Anthimos Xenidis · Alexandros Charitos

Daniel.Reyes-Martinez1@extern.tu-freiberg.de

Table S1. Raw extraction data for LFP selective leaching experiments with industrial black mass. Lithium uncapped to 100%.

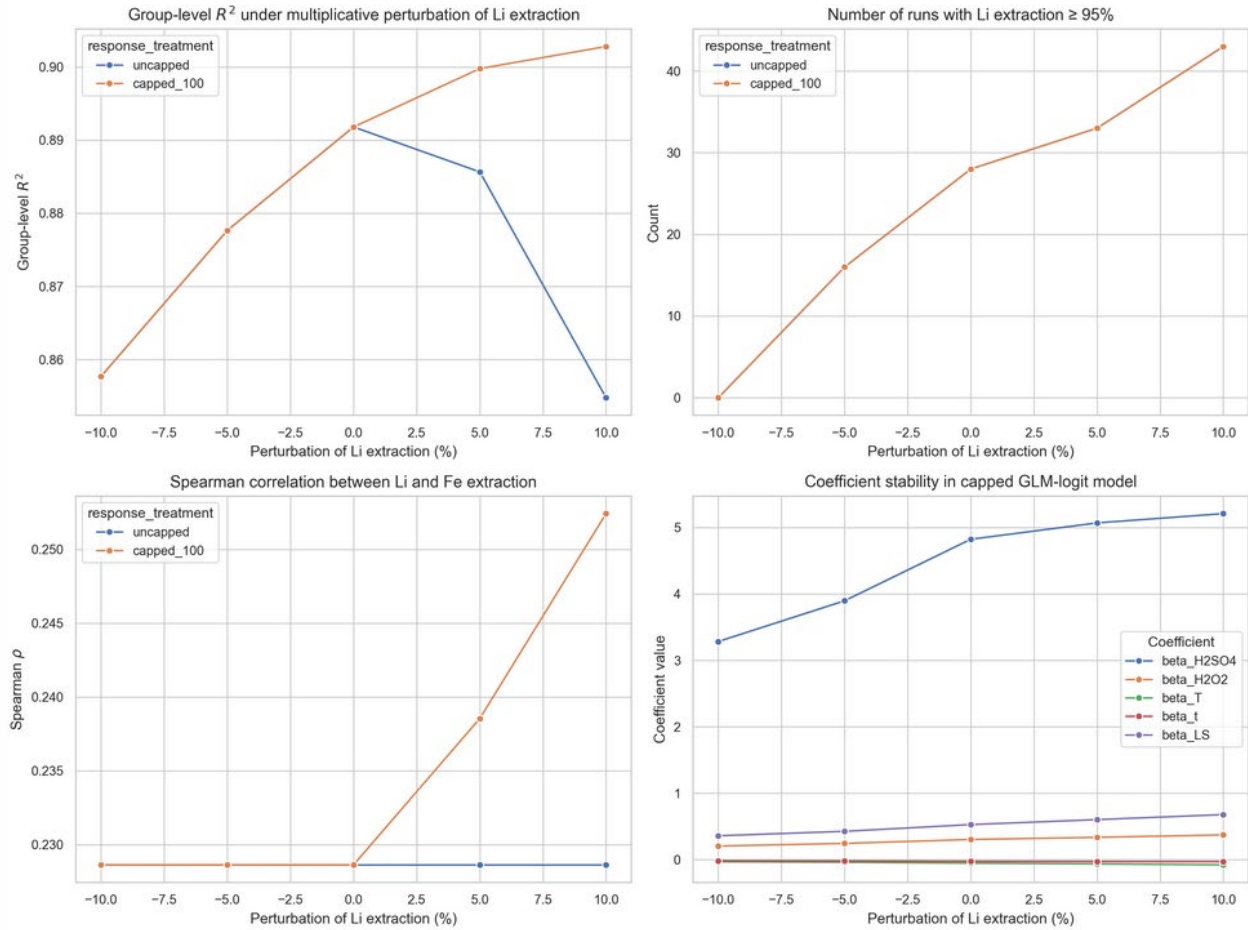
Parameters						Responses, %				
[H ₂ SO ₄]	[H ₂ O ₂]	T (oC)	t (min)	t heating	L/S	Li ext	Fe ext	P ext	Cu ext	Al ext
0.3	1.5	40	30	17	7.5	87.1	1.4	3.9	74.5	56.4
0.3	1.5	40	30	17	7.5	83.4	1.5	3.8	64.3	50.7
0.3	1.5	40	90	17	7.5	79.8	4.2	5.7	64.6	56.5
0.3	1.5	40	90	17	7.5	79.2	2.4	4.3	67.0	57.3
0.4	1.5	40	60	17	7.5	89.0	14.0	14.4	72.6	63.1
0.4	1.5	40	60	17	7.5	110.4	44.7	42.6	90.6	71.5
0.2	1.5	40	60	17	7.5	80.2	0.4	2.7	74.5	48.8
0.2	1.5	40	60	17	7.5	78.9	0.4	2.7	54.9	48.3
0.3	0.6	40	60	17	7.5	74.1	22.8	21.0	62.4	50.7
0.3	0.6	40	60	17	7.5	70.2	15.9	14.9	51.5	42.3
0.3	2.5	40	60	17	7.5	82.1	1.4	3.9	69.5	63.3
0.3	2.5	40	60	17	7.5	85.3	0.9	3.4	59.9	61.7
0.3	1.5	40	60	17	5	58.2	2.2	3.3	40.1	36.9
0.3	1.5	40	60	17	5	54.0	3.2	4.1	40.8	31.8
0.3	1.5	40	60	17	10	79.6	2.6	4.6	66.2	65.2
0.3	1.5	40	60	17	10	99.4	3.1	5.7	98.9	60.2
0.2	0.6	60	60	25	7.5	53.6	9.4	9.0	36.5	35.3
0.2	0.6	60	60	25	7.5	59.7	10.0	9.9	42.4	39.7
0.2	2.5	60	60	25	7.5	56.2	0.1	1.8	40.7	35.7
0.2	2.5	60	60	25	7.5	55.1	0.1	2.0	44.3	36.5
0.4	0.6	60	60	25	7.5	61.2	23.2	21.1	44.7	37.7
0.4	0.6	60	60	25	7.5	60.8	21.4	19.5	54.2	42.9
0.4	2.5	60	60	25	7.5	90.8	0.3	3.0	87.8	55.3
0.4	2.5	60	60	25	7.5	74.0	0.2	2.3	51.8	48.8
0.3	0.6	60	60	25	10	83.7	15.7	15.3	59.6	55.7
0.3	0.6	60	60	25	10	94.0	12.8	13.0	59.4	61.4
0.3	0.6	60	60	25	5	29.9	0.1	1.0	12.8	13.6
0.3	0.6	60	60	25	5	40.0	0.4	1.7	16.8	21.1
0.3	2.5	60	60	25	5	62.7	0.9	3.3	6.8	12.8
0.3	2.5	60	60	25	5	54.5	0.8	2.8	6.0	11.1
0.3	2.5	60	60	25	10	99.0	0.2	3.1	77.3	66.7
0.3	2.5	60	60	25	10	103.5	0.2	3.5	83.3	66.6

0.3	1.5	60	30	25	5	53.7	0.7	2.6	5.8	8.9
0.3	1.5	60	30	25	5	54.3	0.8	2.8	8.1	8.5
0.3	1.5	60	30	25	10	90.6	0.2	2.9	68.4	58.6
0.3	1.5	60	30	25	10	76.0	0.3	2.6	62.9	48.1
0.3	1.5	60	90	25	5	25.3	0.3	1.1	3.1	5.5
0.3	1.5	60	90	25	5	25.3	0.4	1.2	2.6	5.6
0.3	1.5	60	90	25	10	73.1	0.5	2.7	62.8	53.8
0.3	1.5	60	90	25	10	72.9	0.3	2.3	55.4	53.2
0.2	1.5	60	30	25	7.5	71.7	0.1	2.2	53.5	48.1
0.2	1.5	60	30	25	7.5	87.1	0.4	2.8	52.4	55.1
0.2	1.5	60	90	25	7.5	45.4	0.6	2.3	4.1	13.7
0.2	1.5	60	90	25	7.5	47.5	0.7	2.5	4.3	12.3
0.4	1.5	60	30	25	7.5	89.6	1.0	3.4	71.1	61.6
0.4	1.5	60	30	25	7.5	92.2	0.3	3.0	64.5	54.2
0.4	1.5	60	90	25	7.5	49.4	0.1	1.6	2.1	31.8
0.4	1.5	60	90	25	7.5	63.4	0.2	2.0	6.1	42.1
0.2	1.5	60	60	25	5	22.8	0.4	1.5	12.8	5.4
0.2	1.5	60	60	25	5	22.5	0.4	1.7	13.0	5.5
0.2	1.5	60	60	25	10	68.8	0.9	3.7	34.4	11.4
0.2	1.5	60	60	25	10	66.6	0.8	0.3	31.0	12.1
0.4	1.5	60	60	25	10	85.1	0.5	3.5	17.9	58.7
0.4	1.5	60	60	25	10	85.4	0.4	3.2	14.2	60.2
0.3	0.6	60	30	25	7.5	78.2	16.4	15.7	55.2	45.2
0.3	0.6	60	30	25	7.5	78.0	19.0	17.9	58.2	46.9
0.3	0.6	60	90	25	7.5	53.0	0.0	1.5	13.2	17.5
0.3	0.6	60	90	25	7.5	62.3	0.0	1.7	16.3	23.6
0.3	2.5	60	30	25	7.5	73.5	0.7	2.9	49.4	51.9
0.3	2.5	60	30	25	7.5	81.5	1.0	3.7	59.2	61.1
0.3	2.5	60	90	25	7.5	61.9	0.4	2.6	11.4	13.5
0.3	2.5	60	90	25	7.5	60.5	0.3	2.3	21.3	17.1
0.3	1.5	60	60	25	7.5	72.7	0.6	2.8	15.1	10.1
0.3	1.5	60	60	25	7.5	66.4	0.5	3.0	18.0	13.6
0.3	1.5	60	60	25	7.5	64.6	1.0	3.6	8.4	14.9
0.3	1.5	60	60	25	7.5	63.2	1.0	3.6	7.0	13.4
0.3	1.5	60	60	25	7.5	68.3	0.4	2.8	14.6	12.8
0.3	1.5	60	60	25	7.5	72.2	0.6	3.1	10.0	9.4
0.3	1.5	60	60	25	7.5	62.4	1.0	3.7	6.7	12.2
0.3	1.5	60	60	25	7.5	63.4	0.8	3.1	6.1	8.0
0.3	1.5	60	60	25	7.5	61.8	0.8	3.2	5.3	9.8
0.3	1.5	60	60	25	7.5	62.5	0.8	3.2	5.2	9.8
0.3	1.5	60	60	25	7.5	70.5	1.0	3.7	5.8	11.5
0.3	1.5	60	60	25	7.5	64.1	0.8	3.1	5.0	8.9
0.3	1.5	60	60	25	7.5	70.2	0.9	3.2	5.5	10.5
0.3	1.5	60	60	25	7.5	61.6	0.8	2.9	4.7	14.2
0.3	1.5	25	30	0	7.5	98.1	0.6	4.1	11.1	15.4
0.3	1.5	25	30	0	7.5	103.3	1.0	4.6	11.1	14.5

0.3	1.5	25	30	0	7.5	101.4	1.5	5.4	12.2	22.0
0.3	1.5	25	30	0	7.5	104.1	1.5	5.3	10.1	24.7
0.3	1.5	25	90	0	7.5	93.8	0.7	3.6	14.6	17.4
0.3	1.5	25	90	0	7.5	79.0	0.7	3.6	14.6	19.2
0.2	1.5	25	60	0	7.5	78.3	0.6	3.2	63.1	35.6
0.2	1.5	25	60	0	7.5	86.6	0.5	3.4	74.1	36.7
0.4	1.5	25	60	0	7.5	115.9	5.4	9.4	97.3	62.2
0.4	1.5	25	60	0	7.5	64.1	1.7	4.0	58.0	29.7
0.3	0.6	25	60	0	7.5	76.6	16.9	17.6	51.6	35.0
0.3	0.6	25	60	0	7.5	84.1	16.7	17.6	54.6	35.7
0.3	2.5	25	60	0	7.5	78.7	1.0	3.8	65.4	42.6
0.3	2.5	25	60	0	7.5	77.7	0.9	3.7	64.2	43.0
0.3	1.5	25	60	0	5	84.7	1.4	4.6	59.6	43.7
0.3	1.5	25	60	0	5	74.8	1.0	3.8	50.0	39.6
0.3	1.5	25	60	0	10	87.5	2.8	6.1	72.6	45.7
0.3	1.5	25	60	0	10	83.3	2.5	5.5	76.7	41.5

Table S2. Basic statistics for the leaching responses. CV = coefficients of variation.

Response	N	Min	Max	Mean	Median	StdDev	CV(%)	Skewness	Kurtosis
Li	94	25.7	100	80.2	83.7	18.6	23.2	-1.1	0.9
Fe	94	0	48.5	3.9	0.9	7.7	200.4	3.1	11.7
P	94	0.4	46.9	6.0	3.7	6.8	113.7	3.3	13.7
Cu	94	1.4	94.6	27.2	30.5	20.6	75.7	0.7	0.5
Al	94	5.9	78.4	38.0	40.2	22.0	57.8	0.0	-1.4



Treatment: uncapped

Group-level R^2 range: 0.855 to 0.892

Spearman $\rho(\text{Li}, \text{Fe})$ range: 0.229 to 0.229

$\text{Li} \geq 95\%$ count range: 0 to 43

beta_H2SO4: sign(s) = ['+'], relative change vs baseline = -31.9% to 8.0%

beta_H2O2: sign(s) = ['+'], relative change vs baseline = -32.4% to 22.6%

beta_T: sign(s) = ['-'], relative change vs baseline = -39.9% to 49.4%

beta_t: sign(s) = ['-'], relative change vs baseline = -33.8% to 18.9%

beta_LS: sign(s) = ['+'], relative change vs baseline = -31.9% to 28.5%

Treatment: capped_100

Group-level R^2 range: 0.858 to 0.903

Spearman $\rho(\text{Li}, \text{Fe})$ range: 0.229 to 0.252

$\text{Li} \geq 95\%$ count range: 0 to 43

beta_H2SO4: sign(s) = ['+'], relative change vs baseline = -31.9% to 8.0%

beta_H2O2: sign(s) = ['+'], relative change vs baseline = -32.4% to 22.6%

beta_T: sign(s) = ['-'], relative change vs baseline = -39.9% to 49.4%

beta_t: sign(s) = ['-'], relative change vs baseline = -33.8% to 18.9%

beta_LS: sign(s) = ['+'], relative change vs baseline = -31.9% to 28.5%

Figure S1. Sensitivity analysis of the GLM-logit model to multiplicative perturbations of Li extraction and 100% capping of the response variable. Li extraction values were varied by $\pm 5\%$ and $\pm 10\%$ relative to baseline ($\text{Li}' = \text{Li} \cdot (1 + \delta)$), and model performance, $\text{Li} \geq 95\%$ threshold crossings, Li–Fe correlation, and regression coefficients were evaluated for both uncapped and capped ($\leq 100\%$) response formulations.

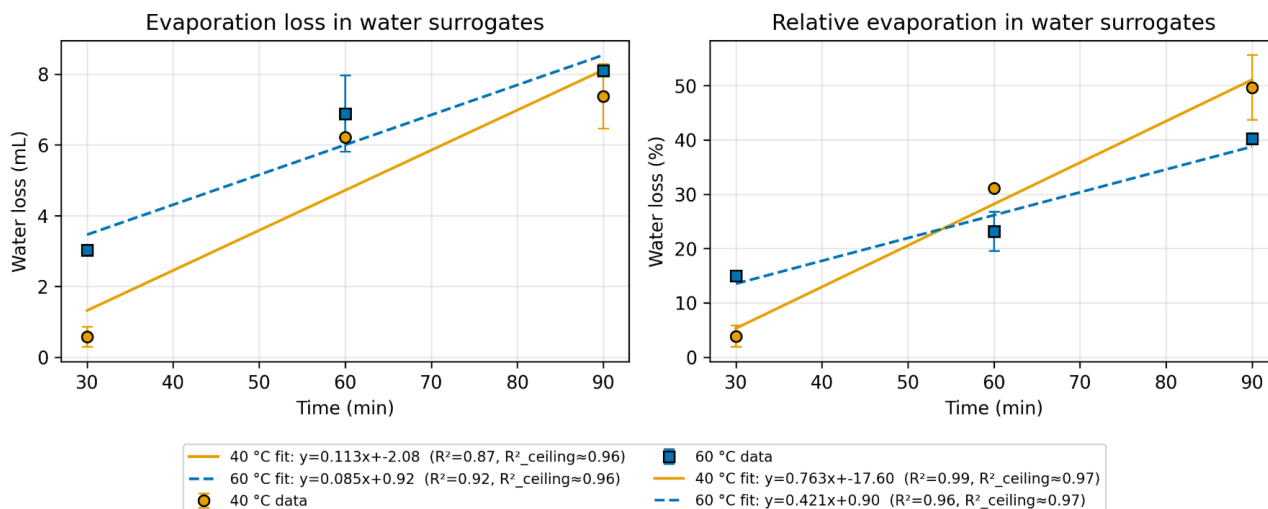
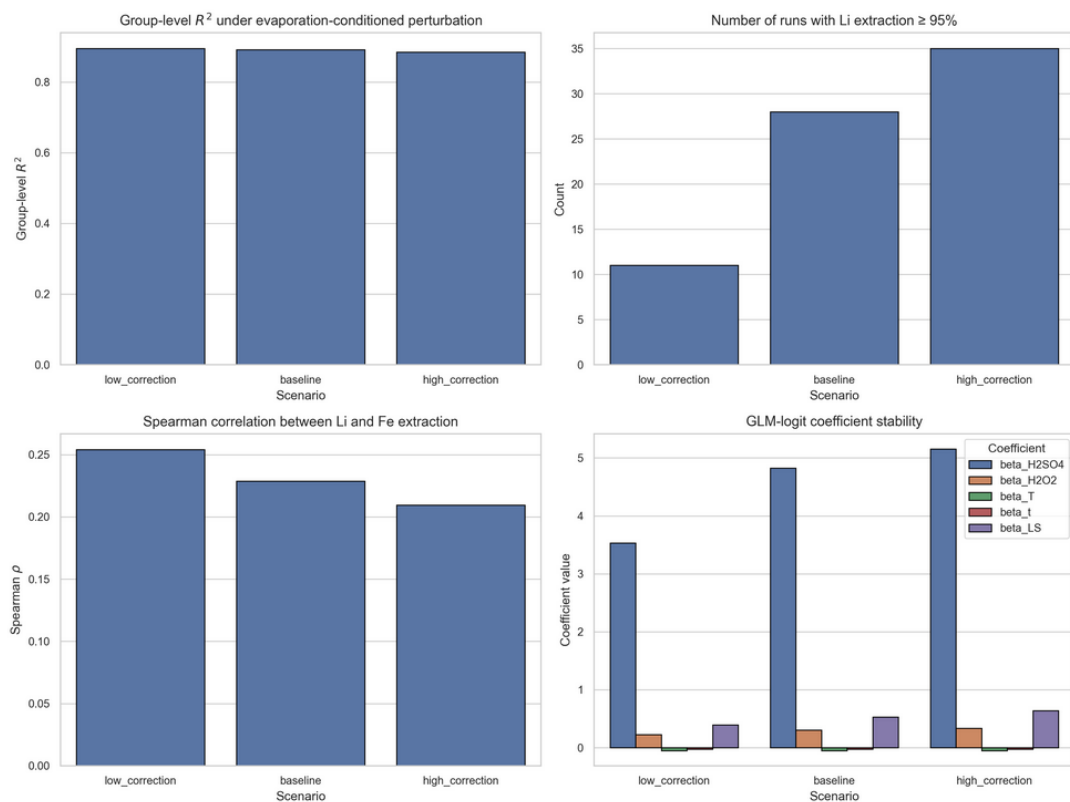


Figure S2. Evaporation data used for correction of solution volumes in extraction calculations. Absolute (left) and relative (right) water loss at 40 °C and 60 °C as a function of time, with linear fits shown. While absolute evaporation increases with temperature, relative water loss at longer times (>60 min) is higher at 40 °C than at 60 °C, indicating that evaporation depends on both temperature and experimental conditions.



low_correction | $R^2(\text{group})=0.895$ | $\text{MSE}(\text{group})=39.11$ | $\text{Li} \geq 95\%=11$ | $\rho(\text{Li,Fe})=0.254$ | signs: $[\text{H}_2\text{SO}_4]=+$, $[\text{H}_2\text{O}_2]=+$, $T=-$, $t=-$, $L/S=+$
baseline | $R^2(\text{group})=0.892$ | $\text{MSE}(\text{group})=40.00$ | $\text{Li} \geq 95\%=28$ | $\rho(\text{Li,Fe})=0.229$ | signs: $[\text{H}_2\text{SO}_4]=+$, $[\text{H}_2\text{O}_2]=+$, $T=-$, $t=-$, $L/S=+$
high_correction | $R^2(\text{group})=0.885$ | $\text{MSE}(\text{group})=38.71$ | $\text{Li} \geq 95\%=35$ | $\rho(\text{Li,Fe})=0.209$ | signs: $[\text{H}_2\text{SO}_4]=+$, $[\text{H}_2\text{O}_2]=+$, $T=-$, $t=-$, $L/S=+$

Figure S31. Sensitivity analysis of evaporation correction effects on group-level R^2 , number of experiments with Li extraction $\geq 95\%$, Li-Fe correlation, and GLM-logit coefficients. Low and high scenarios were generated by applying an evaporation-

conditioned relative perturbation to Li extraction values, defined as $i_{\text{scenario}} = Li_{\text{base}}(1 \pm 0.25 f_{\text{evap}})$, where f_{evap} is the estimated evaporation fraction for each experimental condition.

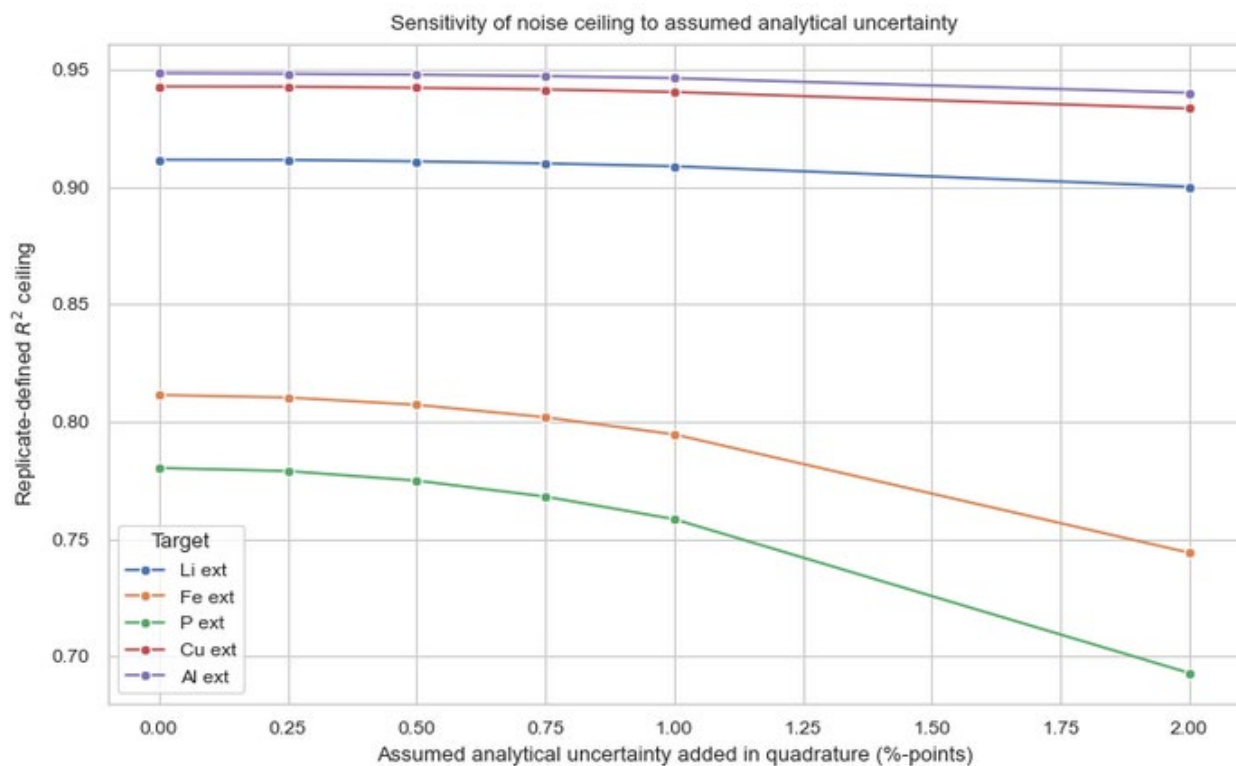


Figure S4. Sensitivity of the replicate-defined R^2 ceiling to the assumed analytical uncertainty. The analytical error floor (0–2 percentage points) was added in quadrature to the within-replicate variance, and the resulting R^2 ceiling was computed as $R^2_{\text{ceiling}} = 1 - \sigma_{\text{rep}}^2 / \sigma_{\text{total}}^2$. Results are shown for all target elements, including the baseline assumption of 0.5 percentage points used in the main text.

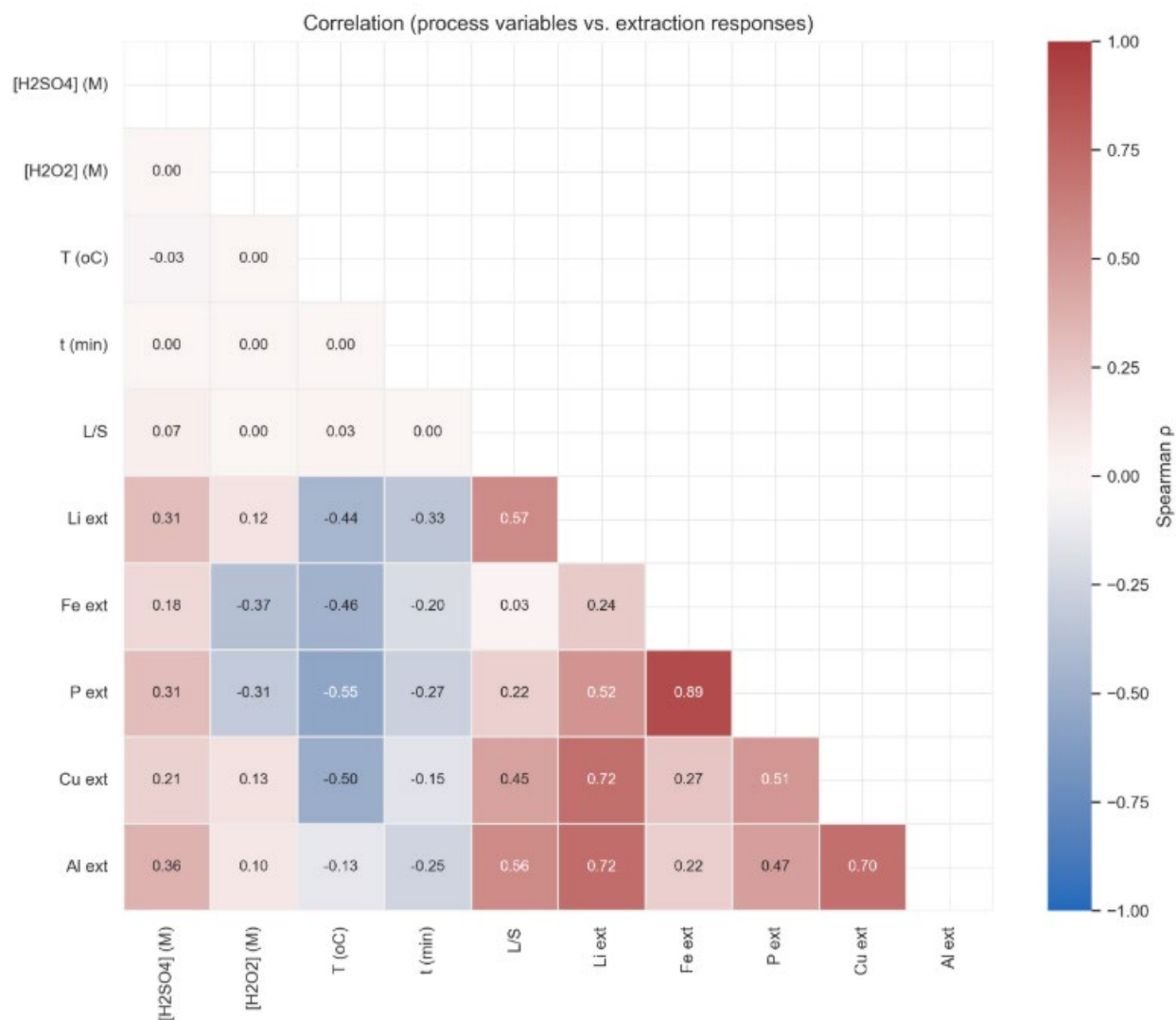


Figure S5. Spearman correlation coefficients between parameters and responses, and between responses. Values near 1 or -1 indicate positive or negative correlation respectively, 0 proximity indicates no correlation.