

FEASIBILITY OF THE CENTRAL COMPOSITE ROTATABLE DESIGN IN COPPER EXTRACTION EXPERIMENTS FROM PRINTED CIRCUIT BOARDS OF TABLETS



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FAPES
FUNDAÇÃO DE AMPARO À PESQUISA DO ESPÍRITO SANTO

Introduction

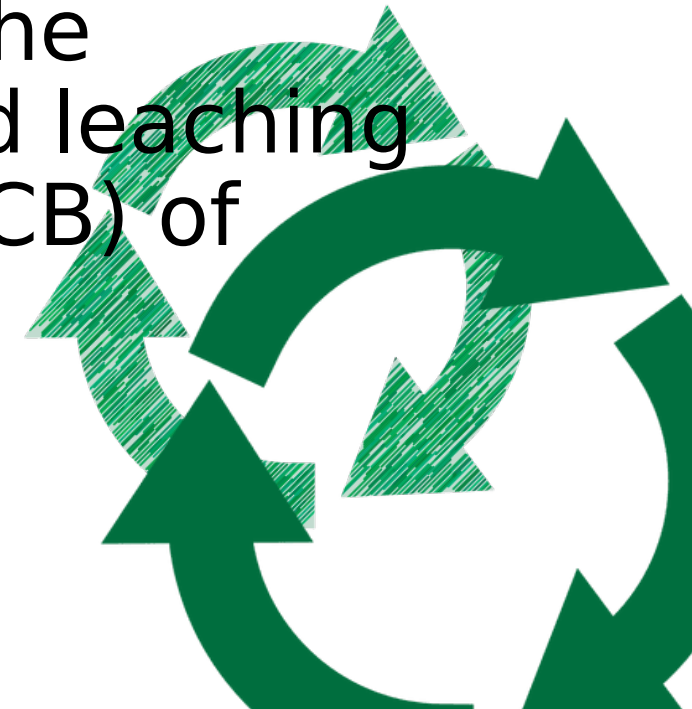
- Unlike other e-waste, tablet waste generation is more recent, and very little is known about its composition and recycling processes
- Tablets entered the Brazilian market in 2010, selling as many as 39,500 units until 2018
- It is estimated amounted to 248 million units worldwide (2015)
- Recycling studies focusing on metals recovery from e-waste usually involves elevated experiments quantity due to combination of all studies variables by Full Factorial Design (FFD) method



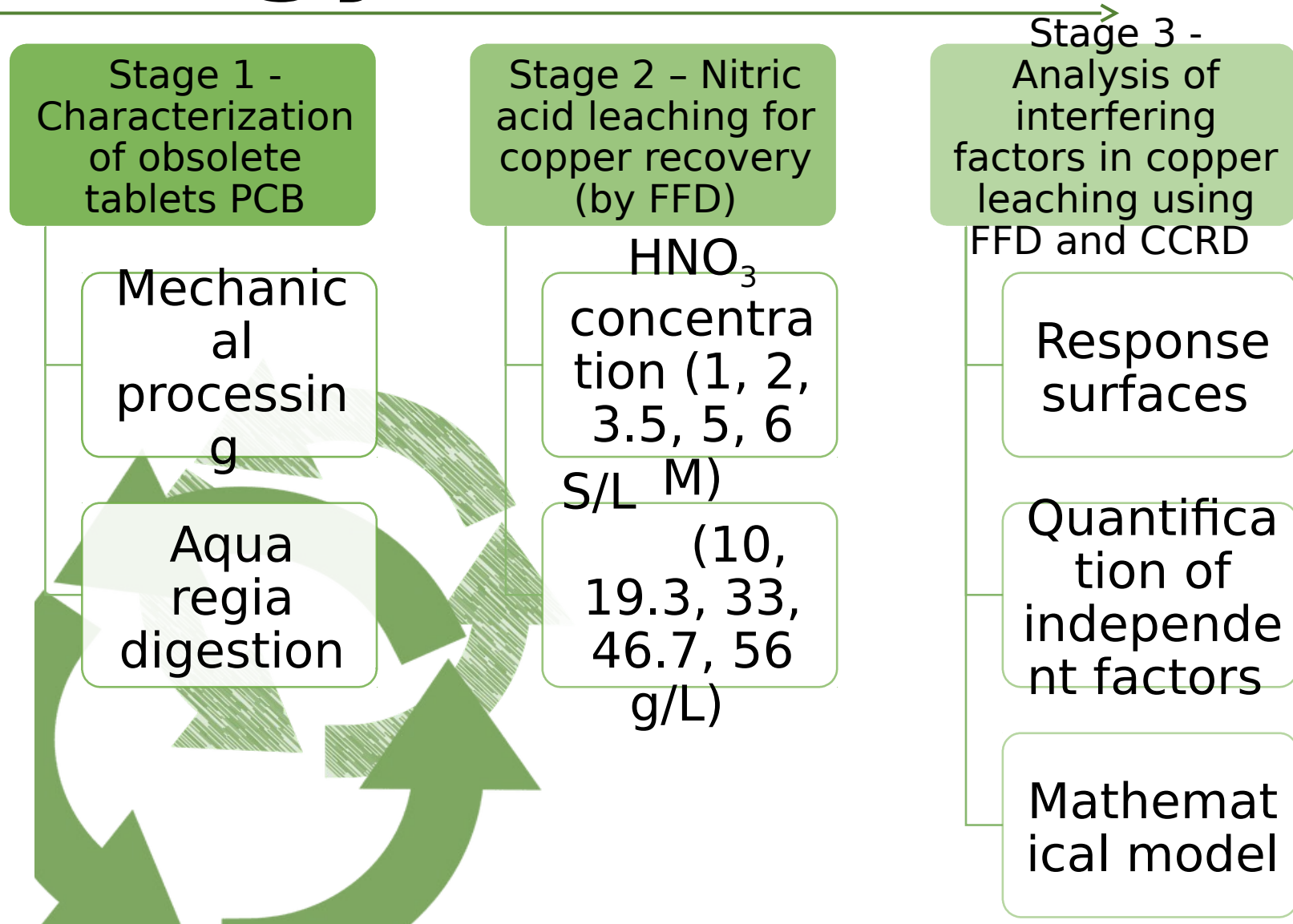
Introduction

- Central Composite Rotatable Design (CCRD) method can be used as an optimized option for the evaluation of e-waste recycling options
- The goal of this study was to evaluate the applicability of the CCRD method in acid leaching of copper from printed circuit boards (PCB) of tablets by analyzing two variables:

Solid liquid ratio and HNO_3 concentration



Methodology



Material and Methods

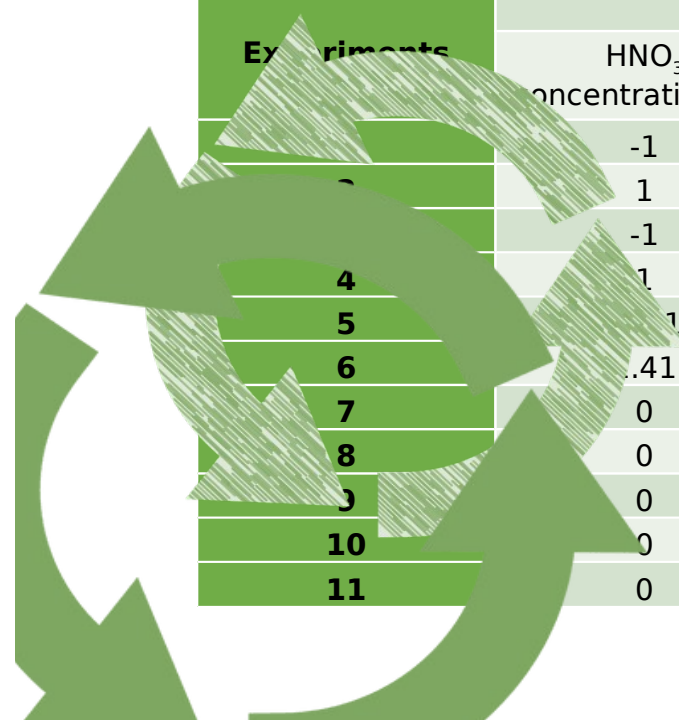


Level values used in the CCRD and FFD for two factors (HNO₃ molar concentrations and S/L ratio) in the nitric acid leaching

Variables Independent	CCRD Levels					FFD Levels				
	-1.41	-1	0	1	1.41	-2	-1	0	1	2
HNO ₃ concentration (M)	1.0	2.0	3.5	5.0	6.0	1.0	2.0	3.5	5.0	6.0
S/L ratio (g/L)	10.0	19.3	33	46.7	56.0	10.0	19.3	33.0	46.7	56.0

Experiments of the CCRD using coded values and applied in the nitric acid leaching

Experiments	Coded values		Applied values	
	HNO ₃ concentration (M)	S/L ratio (g/L)	HNO ₃ Concentration (M)	S/L ratio (g/L)
1	-1	-1	2.0	19.3
2	1	-1	5.0	19.3
3	-1	1	2.0	46.7
4	1	1	5.0	46.7
5	0	0	3.5	33.0
6	1.41	0	6.0	33.0
7	0	-1.41	3.5	10.0
8	0	1.41	3.5	56.0
9	0	0	3.5	33.0
10	0	0	3.5	33.0
11	0	0	3.5	33.0



Material and Methods

Experiments of the Full Factorial Design (FFD) using values coded and applied in the leaching with nitric acid

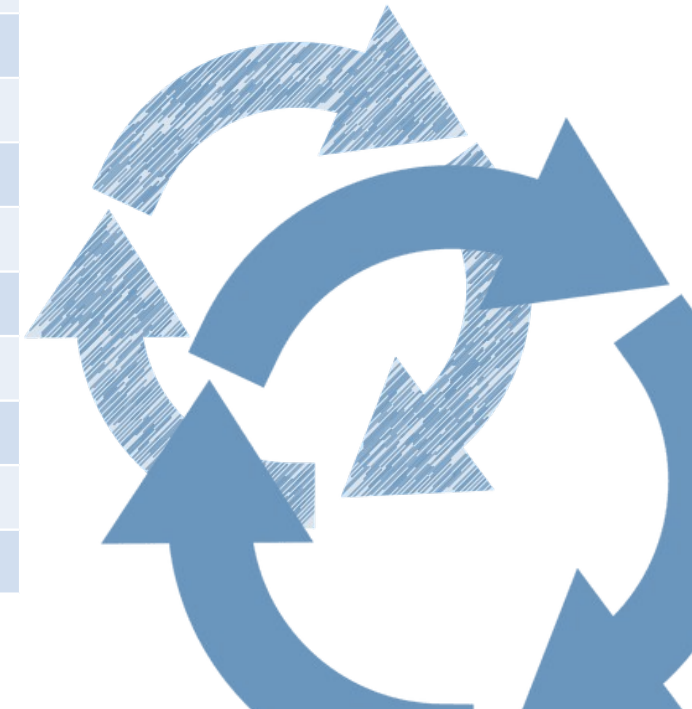
Experiments	Coded values		Applied values	
	HNO ₃ concentration (M)	S/L ratio (g/L)	HNO ₃ concentration (M)	S/L ratio (g/L)
1	-2	-2	1.0	10.0
2	-1	-2	1.0	19.3
3	0	-2	1.0	33.0
4	1	-2	1.0	46.7
5	2	-2	1.0	56.0
6	-2	-1	2.0	10.0
7	-1	-1	2.0	19.3
8	0	-1	2.0	33.0
9	1	-1	2.0	46.7
10	2	-1	2.0	56.0
11	-2	0	3.5	10.0
12	-1	0	3.5	19.3
13	0	0	3.5	33.0
14	1	0	3.5	46.7
15	2	0	3.5	56.0
16	-2	1	5.0	10.0
17	-1	1	5.0	19.3
18	0	1	5.0	33.0
19	1	1	5.0	46.7
20	2	1	5.0	56.0
21	-2	2	6.0	10.0
22	-1	2	6.0	19.3
23	0	2	6.0	33.0
24	1	2	6.0	46.7
25	2	2	6.0	56.0

Results and Discussion

Stage 1 - Characterization of tablets PCB

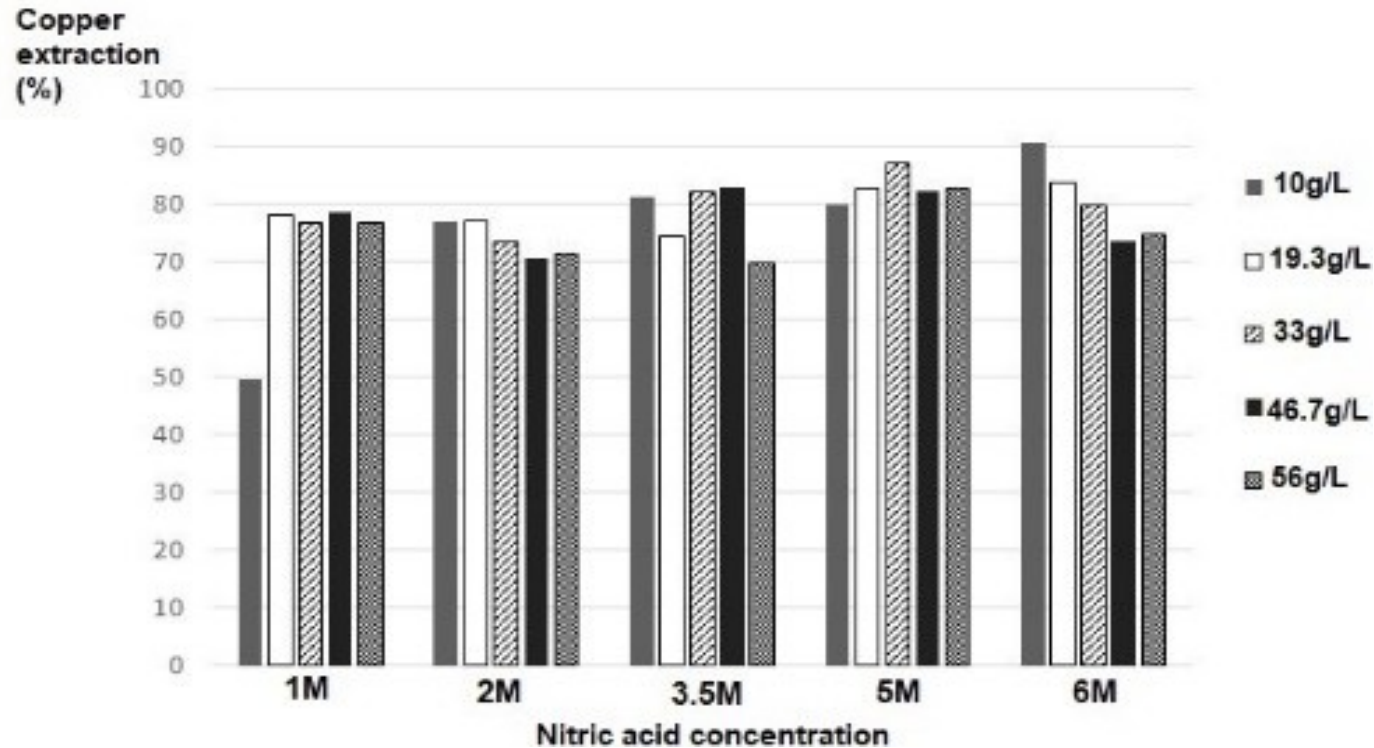
Concentration (wt.%) of the metals from printed circuit boards of tablets

Metals	Metals concentration (wt.%)
Cu	25.76
Sn	3.58
Ba	2.16
Fe	1.62
Ni	1.36
Al	0.69
Zn	0.63
Pb	0.40
Sr	0.04
Au	0.01

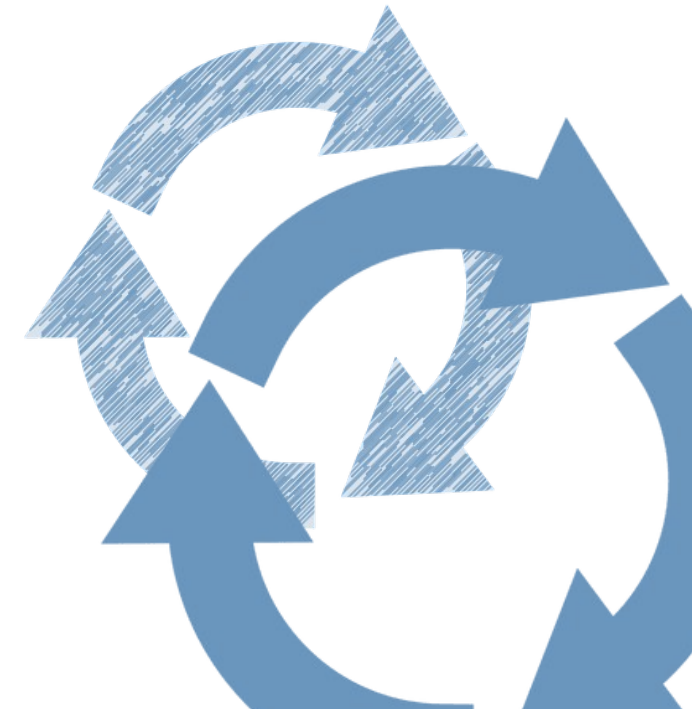


Results and Discussion

Stage 2 - Nitric acid leaching for copper recovery



Copper extraction (%) varying the solid/liquid ratio and the molar concentration of nitric acid (FFD method)



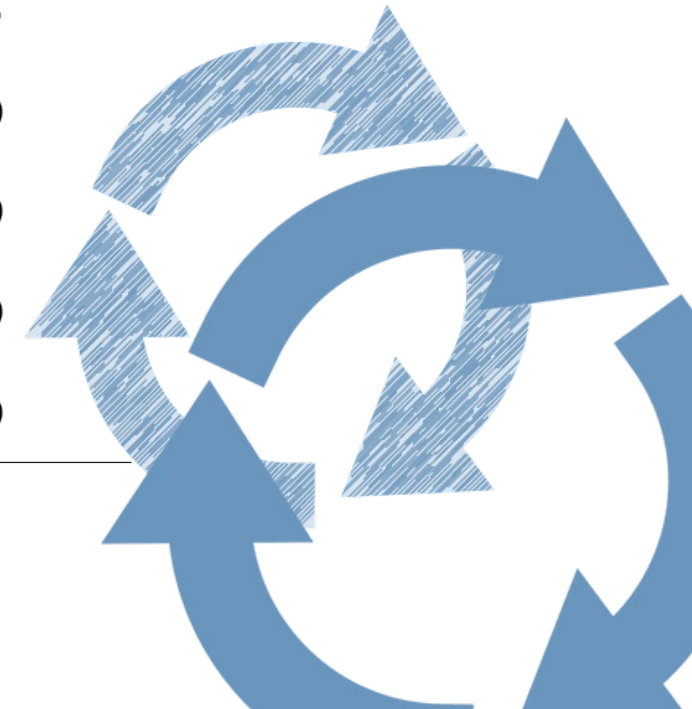
Results and Discussion

Stage 2 - Nitric acid leaching for copper recovery

Results compilation for copper extraction from e-waste by nitric acid leaching

Reference	E-waste	Optimal conditions	Copper extraction (wt.%)
Mecucci & Scott (2002)	PCB	80°C; 6 h; 6M HNO ₃	Above 95
Kinoshita et al. (2003)	PCB	90°C; 6 h; 1M HNO ₃ ; S/L 20g/L	Above 90
Torre & Lapidus (2016)	PCB	25°C; S/L 20g/L; 6h; 0.5M HNO ₃ + 0.1 M H ₂ O ₂ per hour	Above 50
Javed et al. (2018)	AMD Athlon processors	48.89°C; 1h; ultrasound power of 5.52 W; 20% HNO ₃	Above 70
Present study	PCB	60°C; 6h; 1M HNO ₃ ; S/L 56g/L	Above 90

Legend: PCB: printed circuit boards; AMD: advanced micro devices



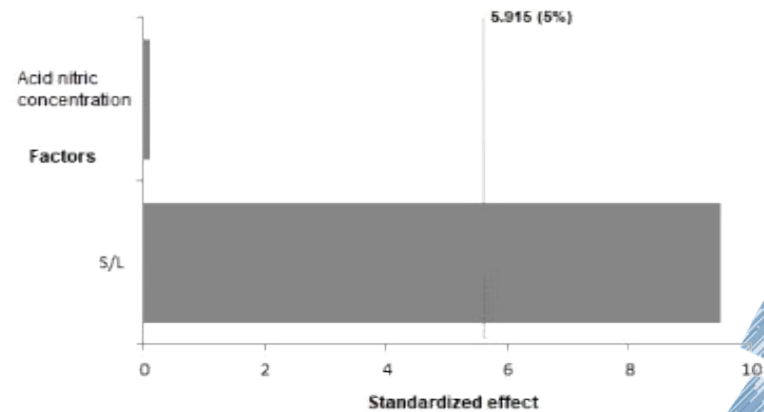
Results and Discussion

Stage 3 - Analysis of interfering factors in copper leaching using FFD and CCRD

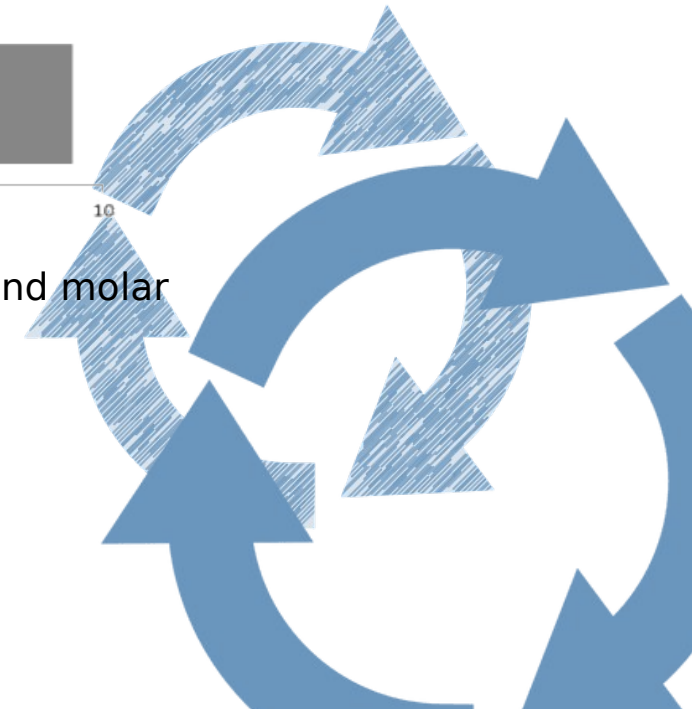
Results used in Central Composite Rotatable Design (CCRD) for two factors

Experiments	HNO ₃ concentration (M)	S/L ratio (g/L)	Extracted copper (%)
1	2	19.3	77.14
2	5	19.3	70.66
3	2	46.7	82.60
4	5	46.7	82.18
5	1	33	81.08
6	6	33	69.66
7	3.5	10	74.45
8	3.5	56	79.68
9	3.5	33	85.09
10	3.5	33	80.33
11	3.5	33	82.71
μ (g)	-	-	78.69
σ (g)	-	-	5.10
C _v (g)	-	-	0.06

Legend: μ = Arithmetic mean, σ = standard deviation, C_v = Coefficient of variation.

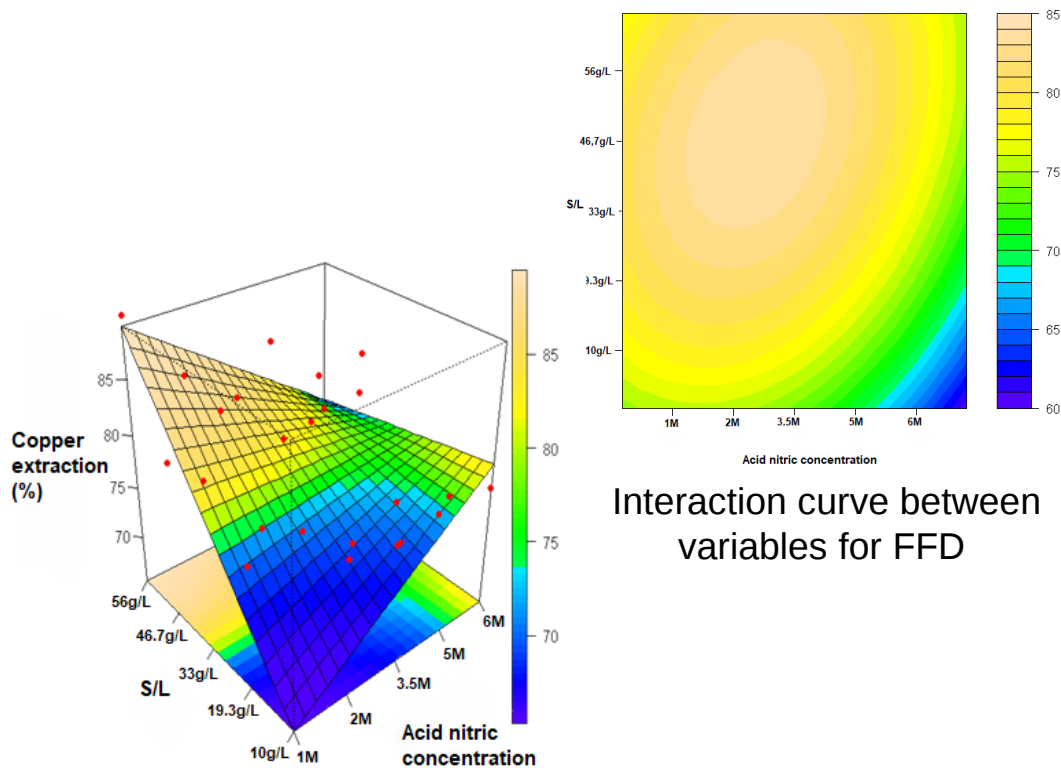


Pareto graphic of the S/L ratio and molar concentration of HNO₃

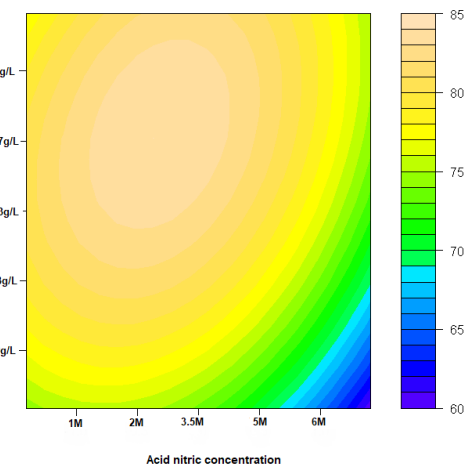


Results and Discussion

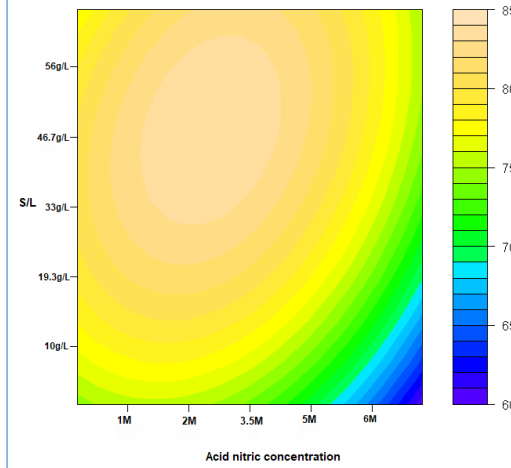
Stage 3 - Analysis of interfering factors in copper leaching using FFD and CCRD



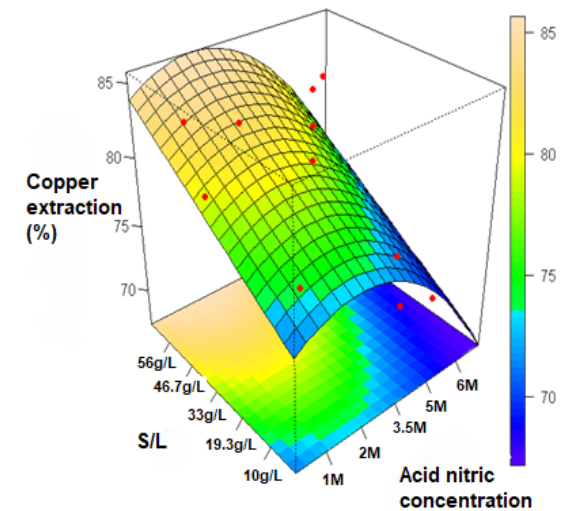
Contour curve for the FFD



Interaction curve between variables for FFD



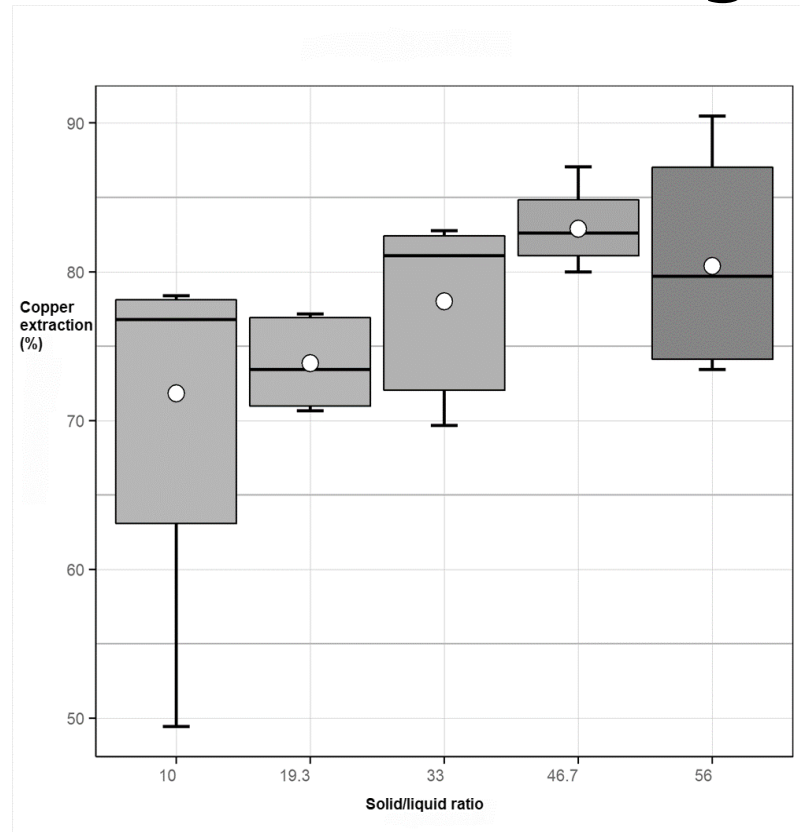
Interaction curve between variables for CCRD



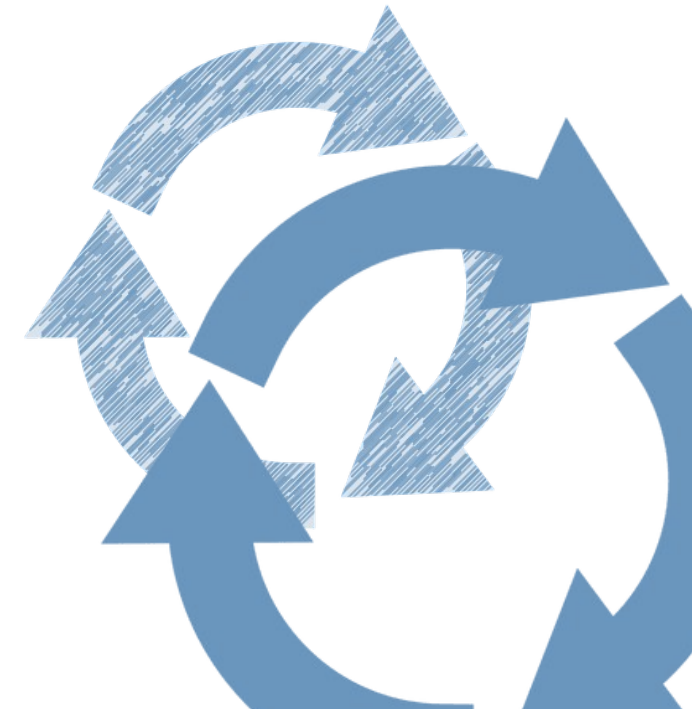
Contour curve for the CCRD

Results and Discussion

Stage 2 - Nitric acid leaching for copper recovery



Boxplot according to solid/liquid ratio



Conclusions

- The concentration of copper (25% wt.) is economically viable from the point of view of recycling, and the presence of gold increases the economic potential, as the tablets are part of the group of new e-waste that create greater interest in recycling, along with smartphones.
- The optimal condition for **90%** copper extraction, as experimentally determined by the FFD method, was 56 g/L (solid/liquid ratio) and 1M nitric acid concentration.
- However, statistical analysis showed that using the CCRD method at 2M concentration of nitric acid and S/L ratio of 46.7 g/L resulted in optimal copper extraction condition reaching **85%** with a much smaller number of tests, which generates input savings and operational time.

Conclusions

- Graphical analysis from Pareto and boxplot showed that the S/L ratio is more influential in copper extraction
- Mathematical models were statistically significant and showed that for the CCRD method the result is within the expected considering the standard deviation
- With only 44% of all FFD method tests, the CCRD method obtained a comparable result
- CCRD method can be potentially applicable to other exploratory studies involving the extraction of metals from printed circuit boards

Acknowledgments

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