The use of amino acids in alkaline environments to recover critical and precious metals from electrical and electronic waste materials

Prof Jacques Eksteen

*Western Australian School of Mines, Minerals, Energy & Chemical Engineering, **Curtin University**, Australia*

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Acknowledgment

Dr Huan Li,
Post-doctoral Fellow, Curtin University

Prof Elsayed Oraby
Associate Professor, Curtin University

Introduction

- Electronic waste can be found in many forms, e.g.: computers, photocopiers, printers, faxes, monitors, batteries and mobile phones
- Worldwide, about 62 million tons/year of E-waste was produced in 2022 and is increasing by around 2.6 million tonnes per year with only around 22.3% of E-waste being collected and recycled
- Australia is one of the world's top ten consumers of electronic devices although its population is fairly small (circa 27 million)
- E-Waste contains valuable resources such as precious metals (Au, Ag, Pd, Pt) and base metals (Cu, Sn, Ni, Zn, Al, Co, Sb). Some be toxic to the environment such as Pb, Hg, and Cd
- Recovery of valuable metals from E-waste can be achieved by using both hydrometallurgical and pyrometallurgical routes

Differences between ores from mine versus e-waste from “urban” mine

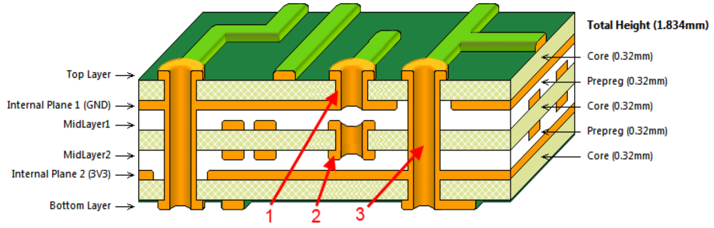
- E-waste is processed in urban / city environments. Mines are normally far from cities.
- E-waste is low density. It cannot be transported far with road transport because it will become uneconomic.
- E-Waste is distributed over wide geographic region (each town generates its own e-waste, where natural mines is a large mineral deposit in one place.
- E-waste is much more difficult to sample representatively / correctly because of the extreme heterogeneity and big difference in particle shapes and densities.
- Smelting has been traditionally been used, but this requires large amounts of low-density material to be shipped to a single location. In a low population density country like Australia, it is much better to process e-waste close to where it is generated. For this Hydrometallurgy is better, because hydrometallurgical plants can be smaller and still be economic.

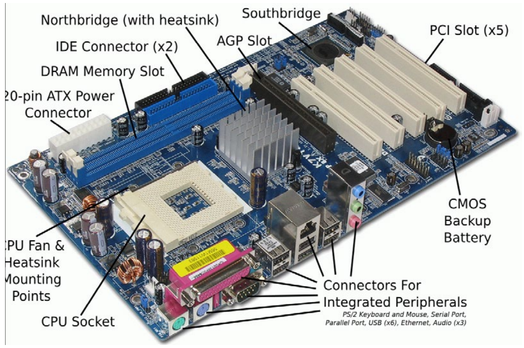
The most representative E-waste: PCB

40% metals

30% plastics

30% ceramics





Normal metals:

Ca, Cu, Fe, Al, Sn, Ba, Zn, Ti, Sb, Ni, Mg, Na, Mn, Sr, K, Bi, Zr, Mo, Co, Se, Li, Ga, V, Hf, Th, U, Be, Te

7.5×10^4 6.2×10^3 1.2×10^3 41 1

Precious metals:

Ag, Au, Pd, Pt

849 5.3

Rare earth metals (REMs):

La, Ce, Sc, Y

91.5 9.7

Heavy metals:

Pb, Cr, As, Cd, Hg

2.4×10^4 1

Non-metals:

Si, C, Cl, B, Br, S, P, F, I

1.4×10^5 200

(All the contents are in ppm)

Source: Li, H., Eksteen, J. and Oraby, E., 2018. Hydrometallurgical recovery of metals from waste printed circuit boards (WPCBs): Current status and perspectives – A review. Resour. Conserv. Recy., 139: 122-139.

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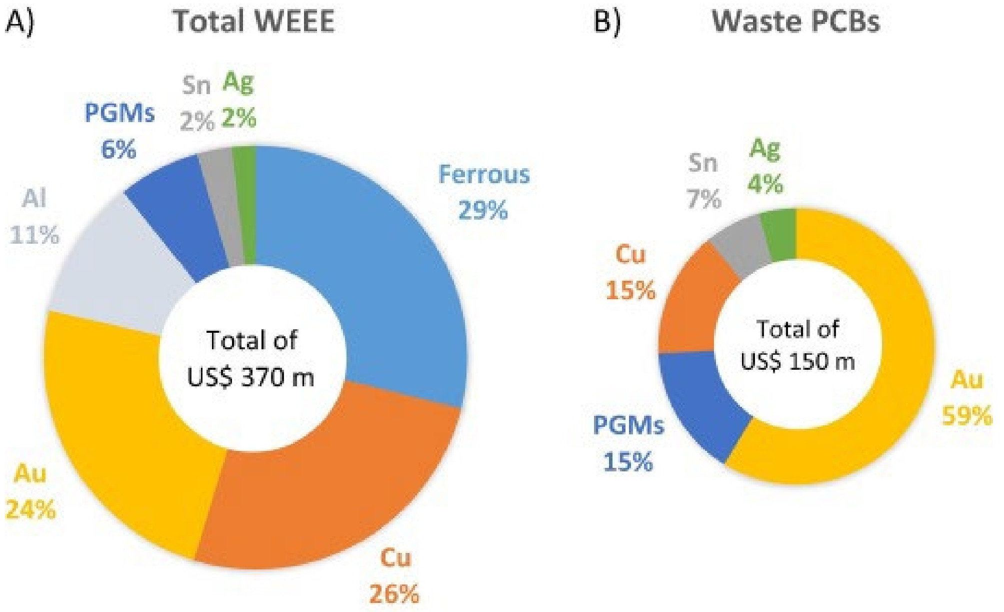
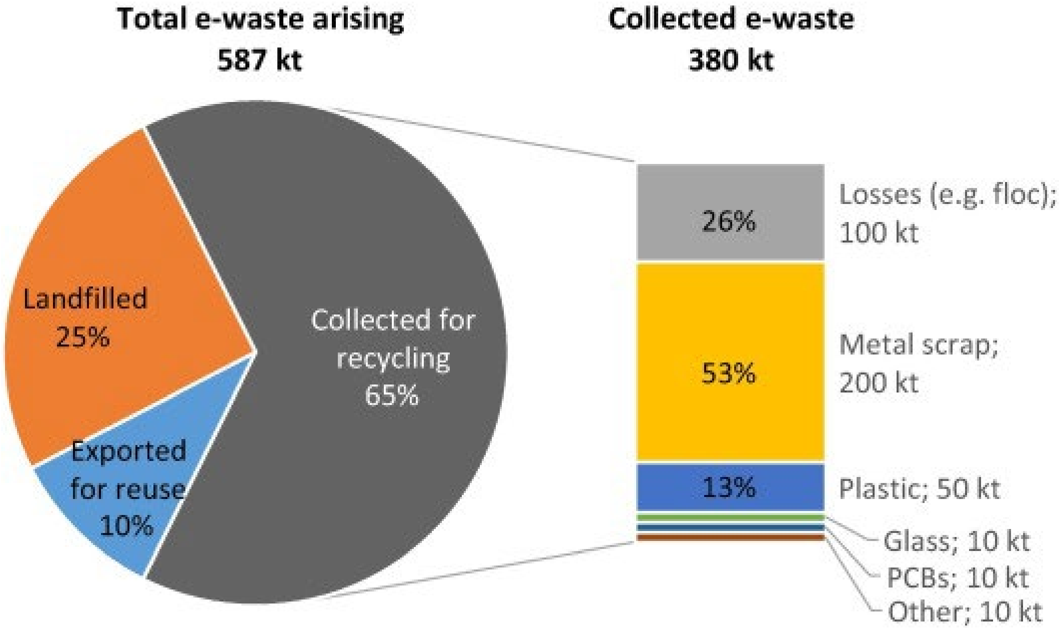
E-waste in Australia



Estimated major metals value in WEEE in Australia in 2014:
(a) total WEEE; (b) waste PCBs



Estimated e-waste destiny in Australia in 2014

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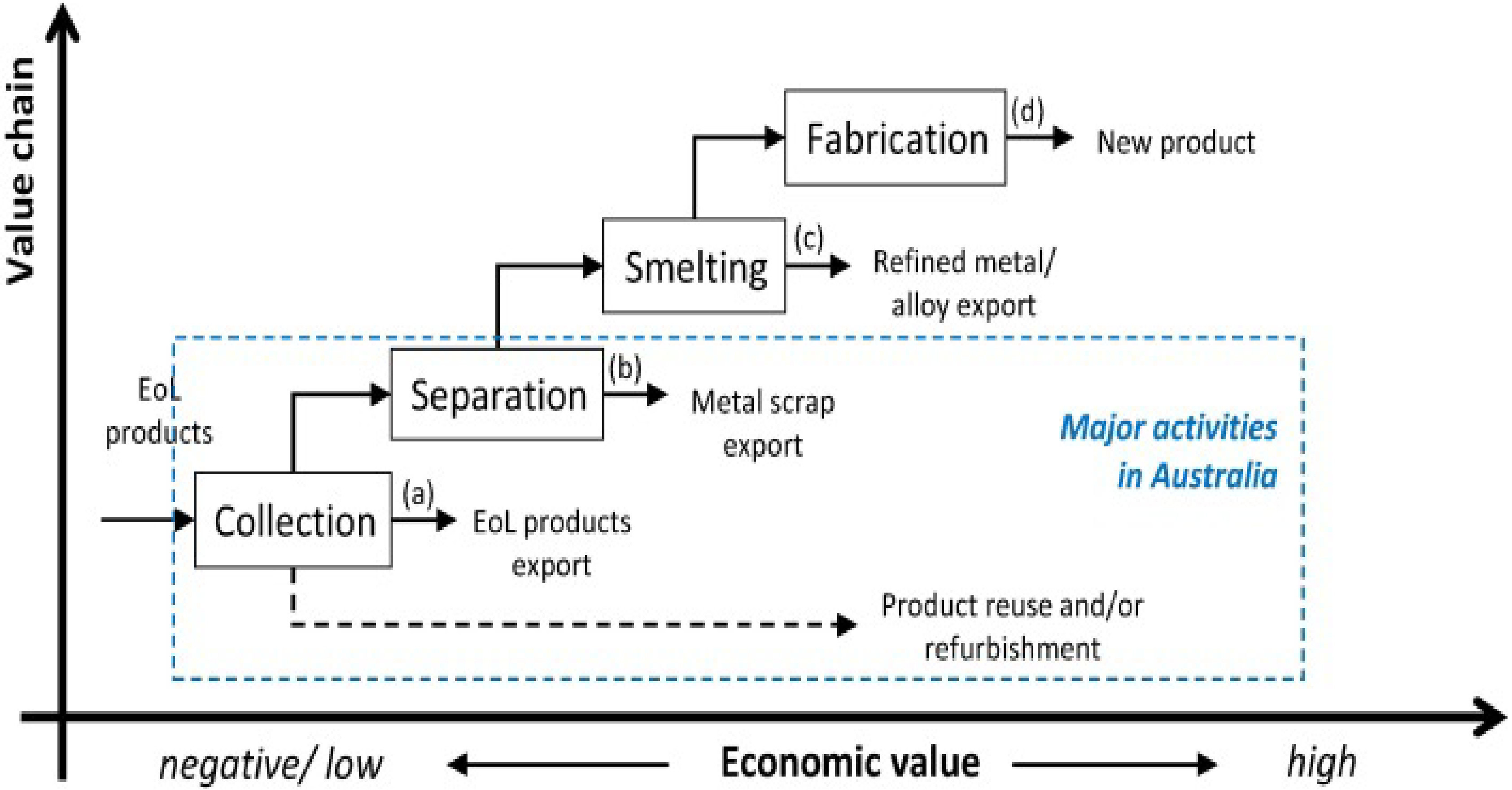
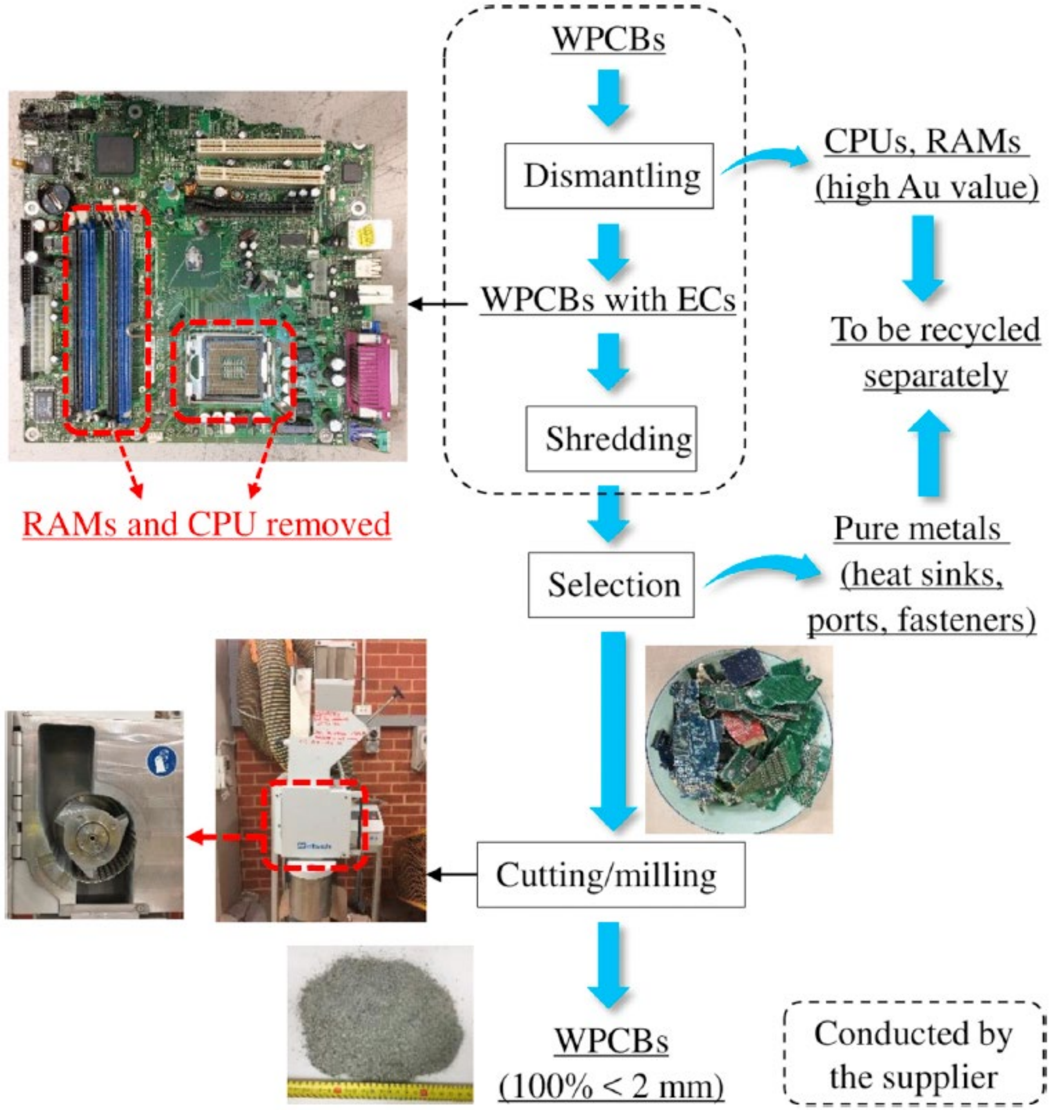
E-waste in Australia: Physical versus chemical processing

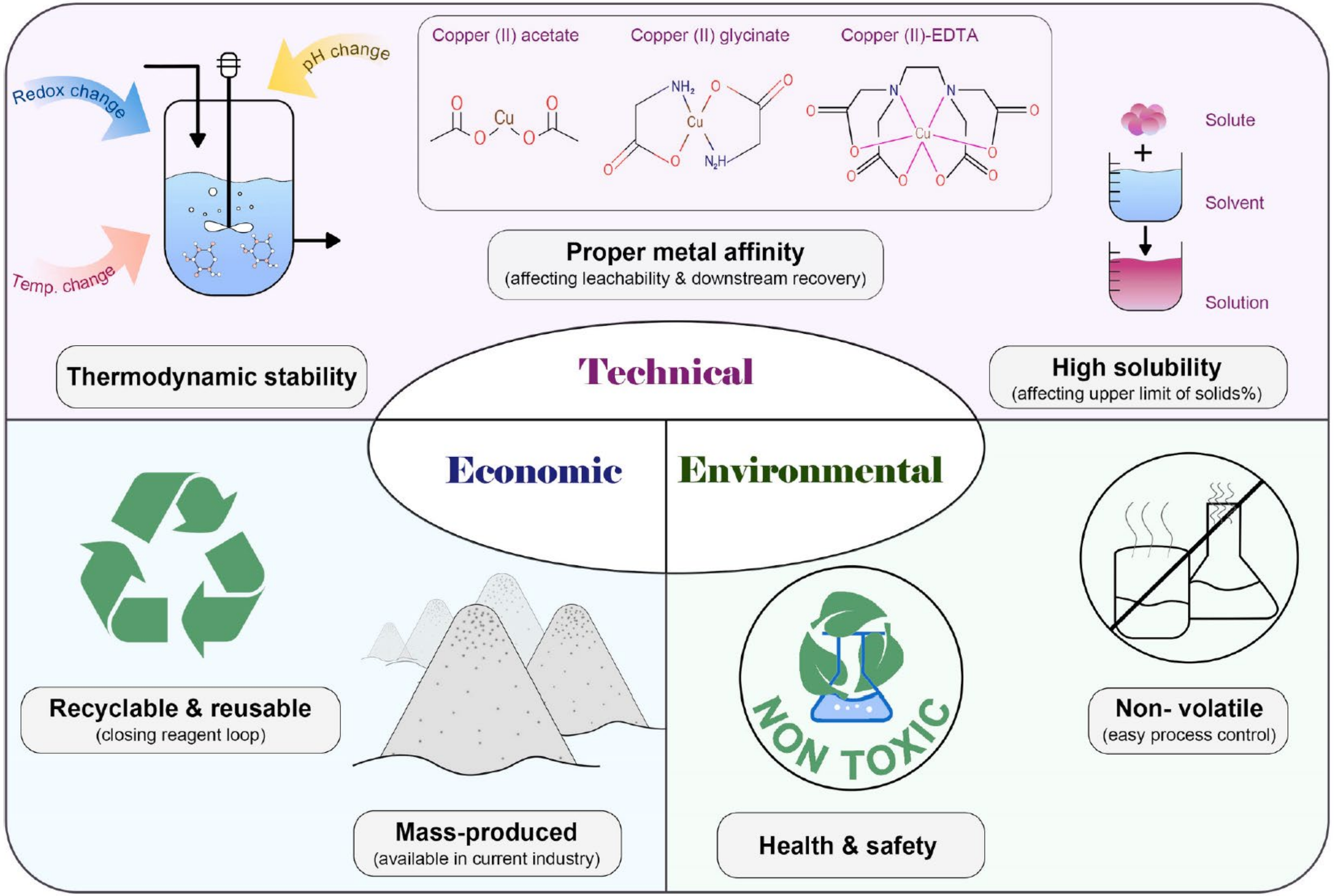


Value chain for EoL products recycling

Scheme of the pre-treatment of WPCBs with electronic components (ECs) mounted



The selection criteria for a green organic ligand for a coordination leaching system



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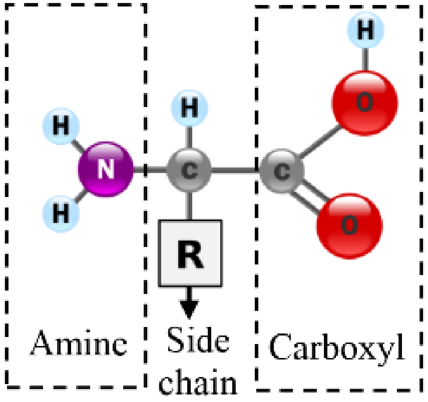
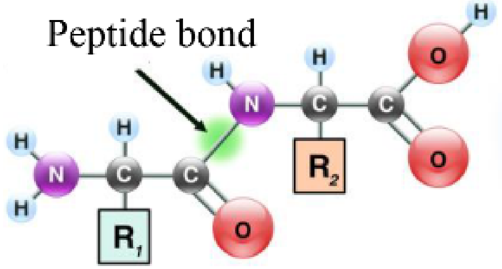
Some prominent α-amino acids used in our research

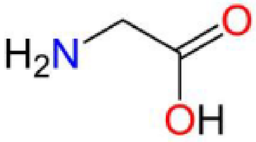
α-amino acid general structure



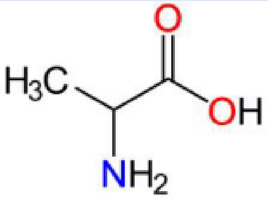
Peptide



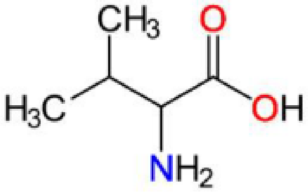
Glycine（Gly）
MW: 75.1 g/mol



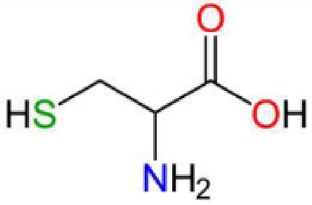
Alanine（Ala）
MW: 89.1 g/mol



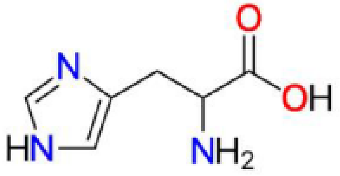
Valine（Val）
MW: 117.2 g/mol



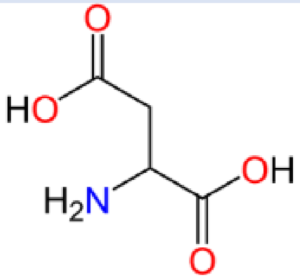
Cysteine（Cys）
MW: 121.2 g/mol



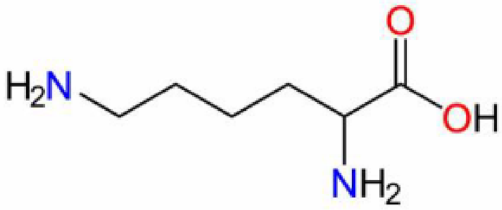
Histidine（His）
MW: 155.2 g/mol



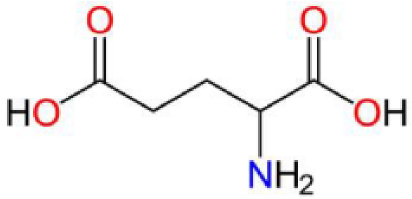
Aspartic acid（Asp）
MW: 133.1 g/mol



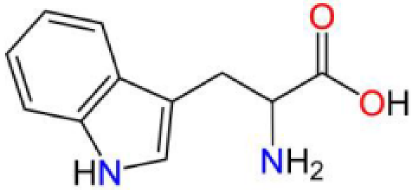
Lysine（Lys）
MW: 146.2 g/mol



Glutamic acid（Glu）
MW: 147.1 g/mol



Tryptophan（Trp）
MW: 204.2 g/mol



Attributes of amino acids (e.g. glycine, glutamic acid) for processing in alkaline environments

Reagent is non-toxic	Reagent recovery and reuse is easy and cost-effective	It can be operated under dilute and concentrated modes
It is environmentally benign	The reagent cost is low (<USD 2000/tonne).	It is chemically stable (compared to cyanide, thiosulfate, etc.).
It has a high affinity for Au, Ag, Cu, Zn, Pb, Pd, Cd, Ni, Co	It can be used synergistic with cyanide.	The precious metal glycinate complexes adsorb well onto activate carbon.
Given the alkaline operation, there is no or very limited interaction with acid consuming materials.	It can be applied in various leach modes (such as heap, in-situ, vat and agitated tank leaching).	The alkaline operation allows low cost materials of construction.
Highly soluble, but non-hygroscopic crystals	No transportability & logistics, trade restrictions	Ease of base metals removal / recovery
Thermally stable	Non-volatile	Simple chemistry
Insignificant Fe, Mn, Mg, Si, Cr dissolution.	Cupric glycinate/glutamate is a good oxidant and acts synergistically with oxygen.	No pH changes required between base and precious metals leaching stages

Research coverage of glycine leaching

Leach System	Reference
Gold	Eksteen & Oraby, 2015 Oraby & Eksteen, 2015a
Gold/silver	Oraby & Eksteen, 2015b
Copper Concentrates	Oraby and Eksteen 2014
Gold-Copper Ores and Concentrates	Oraby and Eksteen, 2014 Eksteen et al., 2018 Oraby, Eksteen and Tanda, 2017 Li, et al., 2022
Polymetallic Ores	Cabri, Wilhelmij, Eksteen, 2017 Aylmore, M., Eksteen, J.J., Wells, M., Jones, M., 2019
Polymetallic e-waste	Oraby, Li, & Eksteen, 2019 Li, Oraby, Eksteen, 2018, 2020, 2021a, 2021b, 2022a, 2022b

Leach System	Reference
Nickel and Cobalt	Oraby et al., 2023a, 2023b Eksteen, Oraby & Nguyen, 2020
Chalcopyrite	Eksteen, Oraby and Tanda, 2017 Tanda et al., 2019 O’Connor and Eksteen, 2020 O’Connor et al., 2018b
Copper oxide minerals	Tanda, Oraby & Eksteen, 2017 Tanda, Oraby & Eksteen, 2021
Chalcocite	Tanda, Oraby and Eksteen, 2018
Zinc and Lead Sulfides	Saba, M., 2019
Copper Metal	O’Connor et al., 2018a; Li, Oraby & Eksteen, 2021b

A summary of various recovery methods for glycine-based leaching system (1)

Methods	Leachates	Optimum conditions	Au, %	Cu, %	Referenc
Solvent extraction	Synthetic Gly-only, Gly:Cu = 3:1, Cu = 2g/L, pH = 10	5% Mextral 84H, A:O = 2:1, RT, 10 min	- [a]	99.90	(Tanda e al., 2017
		10%, Mextral 54–100, A:O = 2:1, RT, 10 min	-	95.87	
	Malachite leachate, Gly-only, Gly:Cu = 4:1, Cu = 2.620 g/L, pH = 12	5% Mextral 84H, A:O = 2:1, RT, 10 min	-	99.93	
		10% Mextral 54–100, A:O = 2:1, RT, 10 min	-	99.39	
	Chalcopyrite leachate, Gly-only, Gly:Cu = 4:1, Cu = 2.288 g/L, pH = 10.5	5% Mextral 84H, A:O = 2:1, RT, 10 min	-	95.57	
		10% Mextral 54–100, A:O = 2:1, RT, 10 min	-	93.20	
	Chalcopyrite leachate, Gly-only, Glycine = 0.1M, Cu = 3.596 g/L, pH = 11.5	10% LIX84-I, A:O =1:1, 40°C, 10min	-	99.4	(Eksteen et al., 2017c)
	E-waste leachate Gly-only, Glycine = 0.2M Cu = 4.18 g/L, pH = 10.6	15% Mextral 84H, A:O = 1:1, RT, 10 min	-	92.32	(Li et al., 2021b)
		30% Mextral 54–100, A:O = 1:1, RT, 10 min	-	87.49	

A summary of various recovery methods for glycine-based leaching system (2)

Methods	Leachates	Optimum conditions	Au, %	Cu, %	Refe
Sulphide precipitation	Chalcopyrite leachate, Gly-only, Glycine = 0.1M, Cu = 1.243 g/L, pH = 11	NaHS powder, HS:Cu = 1:1, RT, 10 min	-	99.1	(Ek et al 201
	Synthetic Gly-only, Gly:Cu = 3:1, Cu = 2g/L, pH = 10	NaHS powder, HS:Cu = 1:1.2, RT, 10 min	-	98.8	(Ta 201
	Synthetic Gly-CN, Gly:CN:Cu = 3:1:1, Cu = 300 mg/L, Au = 1 mg/L, pH = 8-12	5M NaHS solution HS:Cu = 1:1.1 – 1:1.3, RT, 5 min	0	Cu_T: ~68 Cu^{2+}: 100	(De al., 201
	E-waste leachate Gly-only, Glycine = 0.2M Cu = 4.18 g/L, pH = 10.6	5.19M NaHS solution HS:Cu = 1:1.2, RT, 5 min	-	99.5	(Li 202
Carbon adsorption	Synthetic Gly-only, Gly = 1-15 g/L , Au = 2 mg/L, Cu = 40 mg/L, pH =9-12	2 g/L activated carbon, size range = -2.36 + 2.00 mm, RT, 24h	>99.0	15 - 25	(Ta et al 201
	Synthetic Gly-CN, Gly = 5 g/L , Cu:CN=1:1 Au = 2 mg/L, Cu = 300 mg/L, pH = 11	8 g/L activated carbon, size range = -2.36 + 2.00 mm, RT, 24h	99.0	52	(Ta et al 201

A summary of various recovery methods for glycine-based leaching system (3)

Methods	Leachates	Optimum conditions	Au, %	Cu, %	Refe
Ion-exchange	Synthetic Gly-CN, Gly:CN:Cu = 3:1:1, Cu = 300 mg/L, Au = 2 mg/L, pH = 10.5	7.5 g/L MTS9300 resin, RT, 24h	0.81	Cu_T: ~70 Cu^{2+}: 99.1	(De al.,
	Ni-Co leachate, Gly-only Glycine = 46.3 g/L Ni = 1540 mg/L, Co =38.4 mg/L, Cu = 14.5 mg/L	55 g/L MTS9300 resin, RT	-	Ni: 97% Co: 65% Cu: 99%	(Eks et al 202
	Synthetic Gly-CN, Gly:CN:Cu = 3:1:1, Cu = 1000 mg/L, Au = 6 mg/L, pH = 10.5	6 g/L IXOS-AuC resin, RT, 24h	97.2	Cu_T: 7.4 Cu^{2+}: 1.6	(De al., 202
Chemical reduction	E-waste leachate, Gly-only, Glycine = 0.5M Cu = 5.42 g/L, pH = 12.8	3M hydrazine, Cu: hydrazine = 1:1, 40°C , 2h	-	98.6	(Li 202

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From primary to secondary resources processing

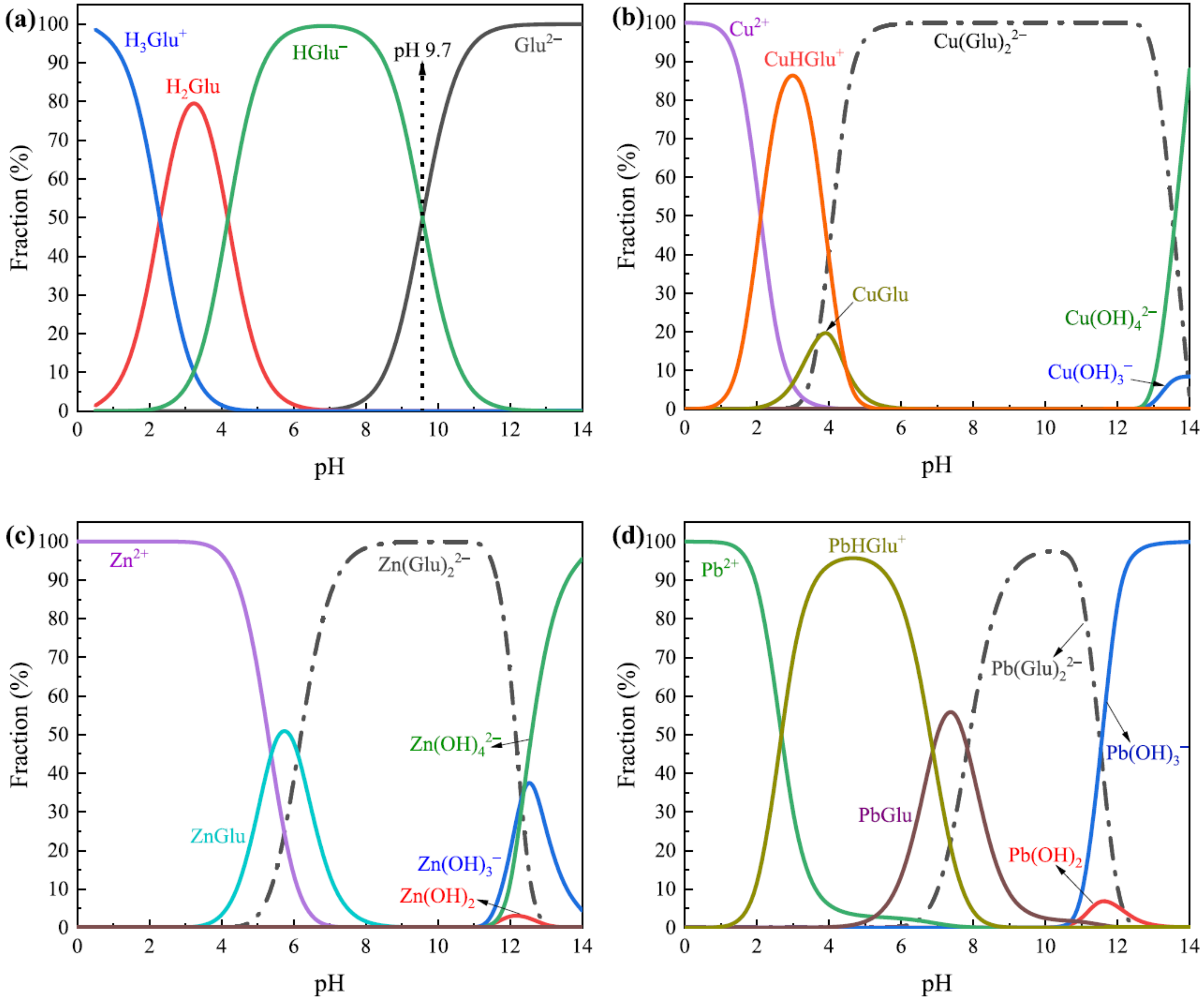
- Since 2012, we have clearly demonstrated, through extensive research, that amino acids, particularly under alkaline conditions can be used to selective leach under mild conditions:
  - Au, Ag, Pd, Pt, Cu, Ni, Co, Cd, Zn and Pb from
  - Ores, tailings, concentrates and process intermediates mineralised as
  - Sulfides, oxides, carbonates and native metals and alloys
- The biggest detracting factors are
  - Leach kinetics
  - Poor ability to solubilise silicate minerals of Cu, Ni and Co (e.g. chrysocolla, olivine and serpentine)
- We have shown that both metal recovery from solution and reagent recovery are feasible and effective for various systems using
  - SX, IX, Precipitation, Crystallisation, Nano-filtration, and carbon adsorption
- The aim of this presentation is to provide leaching examples for E-waste, focussed on waste printed circuit boards

The contents of major metals in the samples of waste PCBs and their economic values (100 % < 2 mm) used in this study

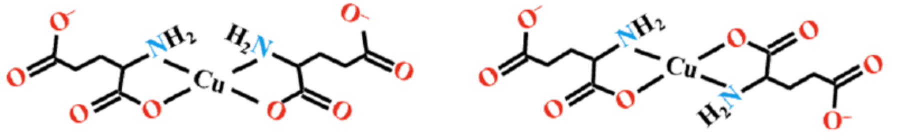
	Content in wt. %							Content in g/t			
	Cu	Fe	Al	Sn	Pb	Zn	Ni	Co	Au	Ag	Pd
Metal price, USD/t	9014	–	2250	29,605	2023	2634	16,250	–	7.87×10^{7}	9.32×10^{5}	3.08×10^{7}
Metal content	22.60	1.83	3.18	2.81	0.34	0.69	865.50	59.72	106.77	170.50	10.37
Economic value [a], %	17.17	–	0.60	7.01	0.06	0.15	0.12	–	70.85	1.34	2.69
Total value [b], USD/t	$11,846.36										

[a] The economic values for the base and precious metals were determined from the official price of metal of the London Metal Exchange (https://www.lme.com/), and The Perth Mint (https://www.perthmint.com/), respectively, on 1st August 2024; [b] The total value was calculated from the metals specified.

Distribution of species of (a) glutamate-H_2O, (b) Cu^{2+}-glutamate-H_2O, (c) Zn^{2+}-glutamate-H_2O and (d) Pb^{2+}-glutamate-H_2O systems as a function of pH

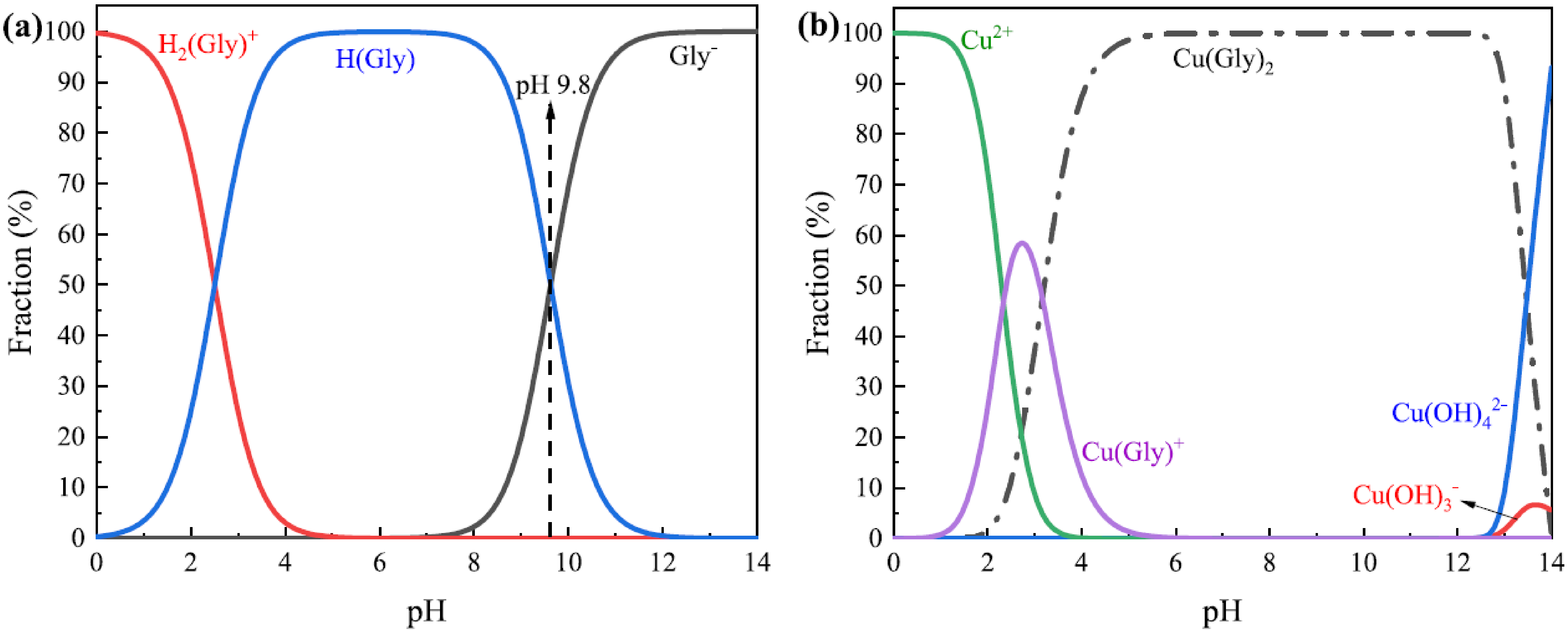


Conditions:
0.5 M glutamate,
50 mM Cu^{2+},
2 mM Zn^{2+},
2 mM Pb^{2+}
T= 25 °C



cis[$Cu(Glu)_2^{2-}$]   *trans*[$Cu(Glu)_2^{2-}$]

Distribution of species of (a) glycine-H_2O and (b) glycine-Cu^{2+} –H_2O systems



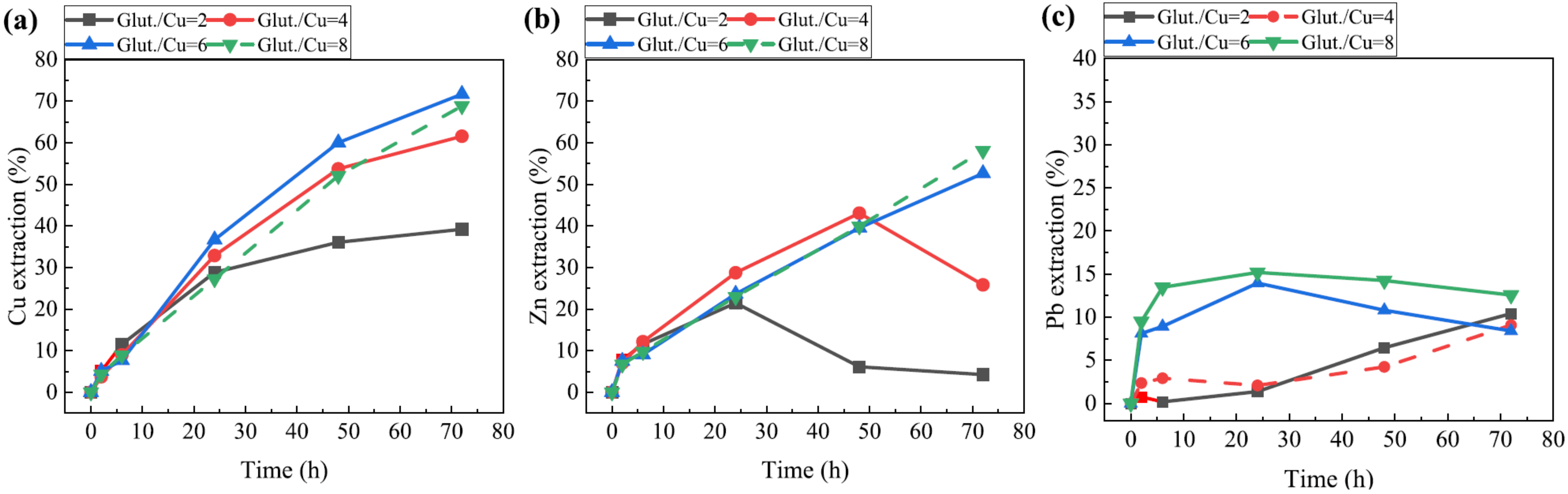
Conditions:
0.5 M glycine,
50 mM Cu^{2+}
T = 25 °C

Cumulative formation constants of the complexes between specified metals and L-glutamic acid, glycine, or ammonia at 20–25 °C

Metal-ion	Cu^{+}	Cu^{2+}	Zn^{2+}	Ni^{2+}	Pb^{2+}	Co^{2+}	Fe^{2+}
Complexing with glycine							
$\log K_{ML}$	–	8.6	5.5	6.2	5.5	5.2	4.1
$\log K_{ML2}$	10.0	15.5	10.0	11.1	10.0	9.3	7.7
$\log K_{ML3}$	–	16.3	–	15.0	–	10.8	–
Ref.	1,2	1,2	1,2	1,2	1,2	1,2	1,2
Complexing with glutamic acid							
$\log K_{ML}$	–	8.2	4.5	5.6	4.6	4.5	–
$\log K_{MLH}$	–	12.4	10.3	–	11.5	–	–
$\log K_{ML2}$	–	14.9	7.8	–	6.8	–	–
Ref.	–	3	3	4	3	4	–
Complexing with ammonia							
$\log K_{ML}$	5.9	4.3	2.4	2.8	–	2.1	1.4
$\log K_{ML2}$	10.9	8.0	4.8	5.0	–	3.7	2.2
$\log K_{ML3}$	–	11.0	7.3	6.8	–	4.8	–
$\log K_{ML4}$	–	13.3	9.5	8.0	–	5.6	–
Ref.	5	5	5	5	–	5	5

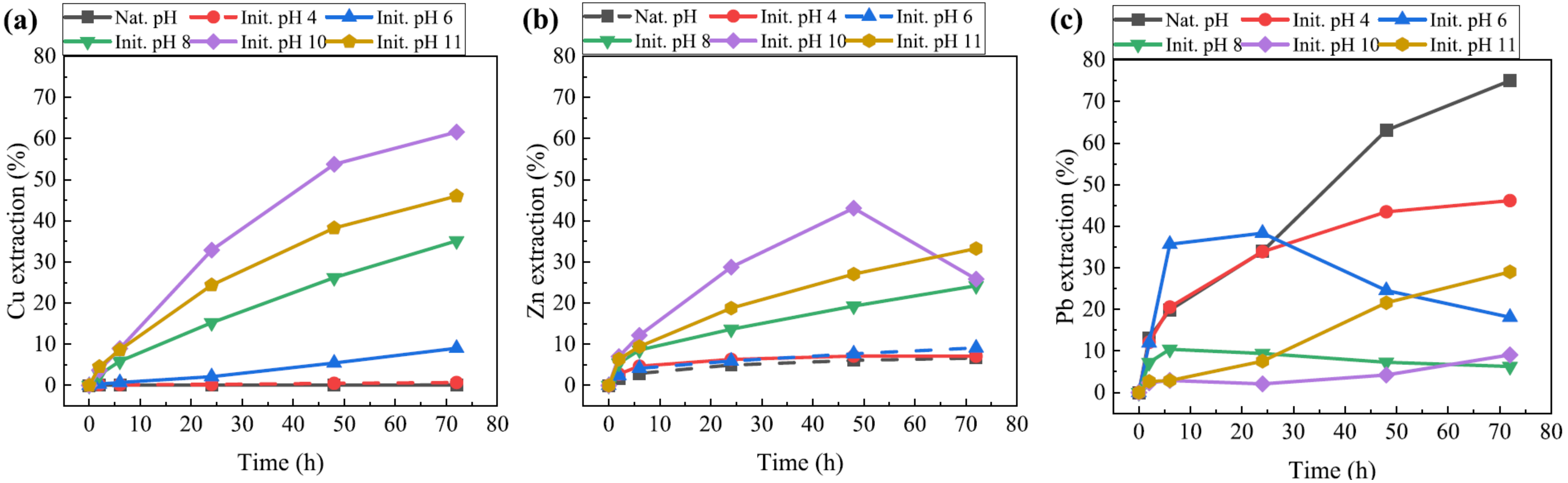
M refers to the metal ion, and ***L*** refers to ammonia, glycinate, or glutamate ligand.

Crushed PCB Leaching:
Effect of glutamate/Cu molar ratio on the extraction of (a) Cu, (b) Zn and (c) Pb



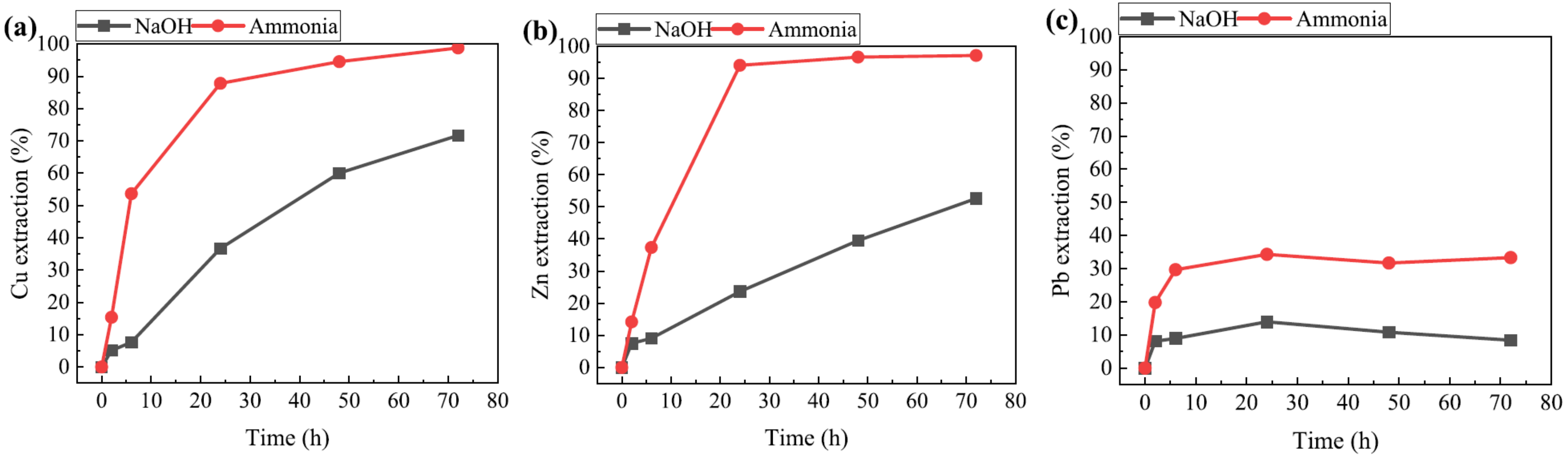
Conditions:
Room Temperature,
2% solids,
Initial pH 10

Crushed PCB Leaching:
Effect of initial pH on the extraction of (a) Cu, (b) Zn and (c) Pb



Conditions:
Room Temperature ,
2% solids,
glutamate/Cu molar ratio= 4

Crushed PCB Leaching:
Effect of pH modifier on the extraction of (a) Cu, (b) Zn and (c) Pb



Conditions:

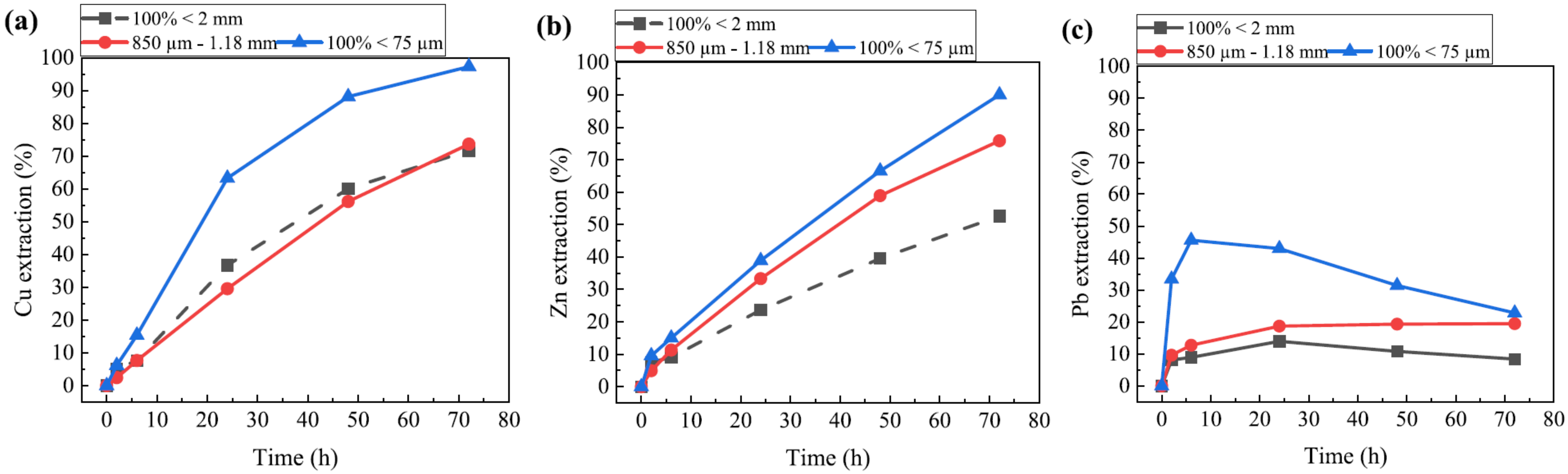
Room Temperature,

2% solids,

Initial pH 10,

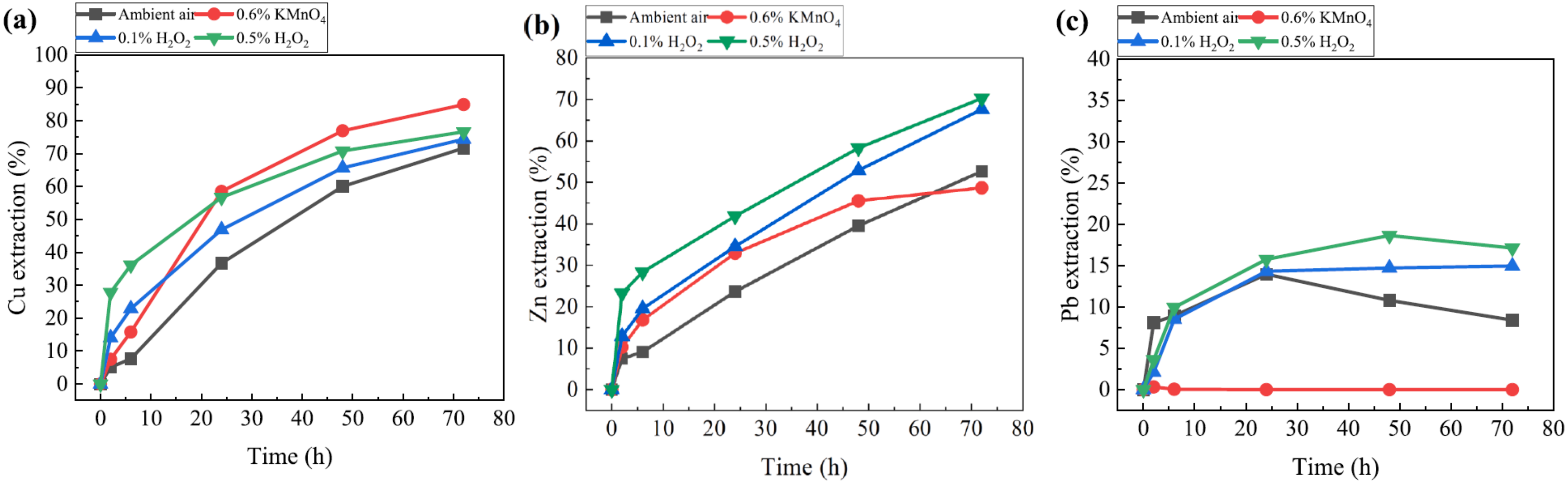
Glutamate/Cu molar ratio = 6

Crushed PCB Leaching:
Effect of particle size on the extraction of (a) Cu, (b) Zn and (c) Pb



Conditions:
Room Temperature,
2% solids,
Initial pH 10,
Glutamate/Cu molar ratio = 6

Crushed PCB Leaching:
Effect of oxidant on the extraction of (a) Cu, (b) Zn and (c) Pb



Conditions:
Room Temperature,
2% solids,
Initial pH 10,
Glutamate/Cu molar ratio = 6

$$MnO_4^- + 2H_2O + 3e^- = MnO_2 + 4OH^-, \ E^\circ = 0.60\ V$$

$$H_2O_2 + 2H^+ + 2e^- \rightarrow 2H_2O, \ E^\circ = 1.76\ V$$

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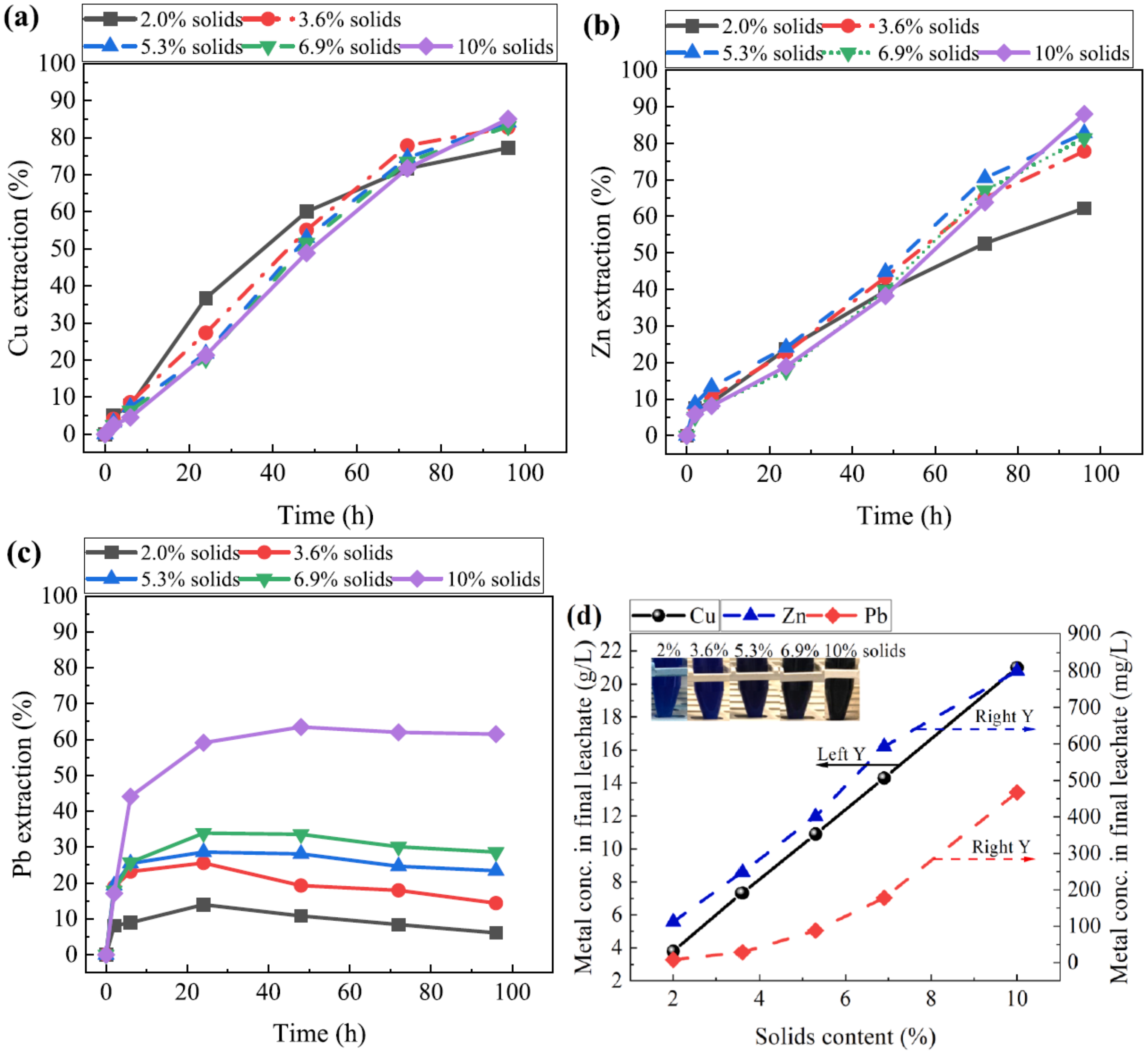
Crushed PCB Leaching: Effect of solids content on the extraction of (a) Cu, (b) Zn and (c) Pb, and (d) concentration of Cu, Zn and Pb in the final leachate



Conditions:
Room Temperature,
Initial pH 10,
Glutamate/Cu molar ratio = 6

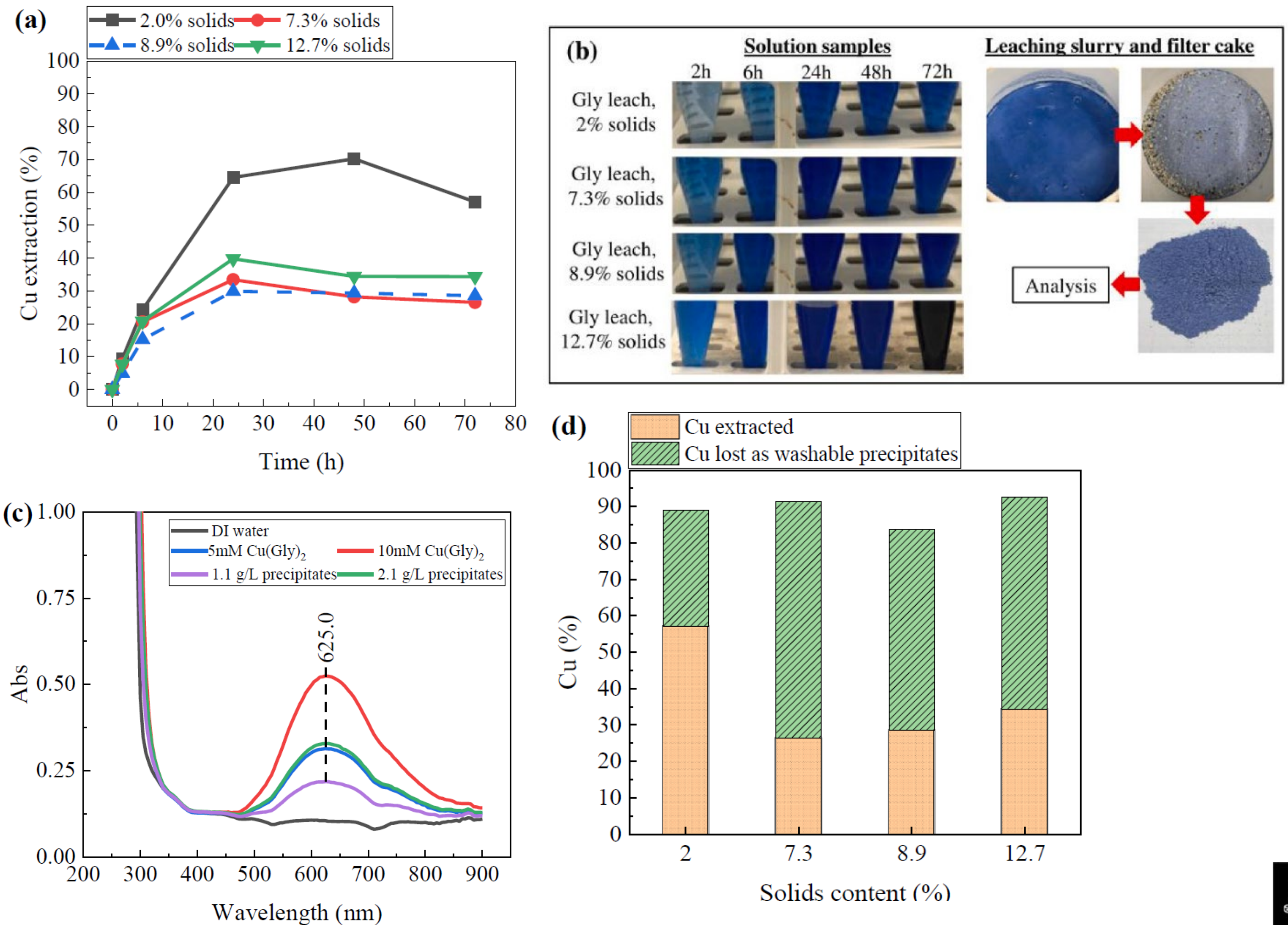
Metals extraction (using glutamic acid) and standard deviation of the triplicated experiments

	Test 1	Test 2	Test 3	Mean	Standard deviation
Cu extraction (%)	88.0	88.0	86.9	87.6	0.51
Zn extraction (%)	87.8	87.1	88.0	87.7	0.39
Pb extraction (%)	65.8	64.1	63.5	64.5	0.98

Extraction of major and other metals under the recommended conditions (RT, 10% solids, initial pH 10, glutamate/Cu molar ratio 6)

	Base metals								Precious metals		
	Cu	Zn	Pb	Al	Co	Ni	Fe	Sn	Au	Ag	Pd
Extraction, %	85.1	88.0	61.5	40.5	11.9	8.9	0.47	5.1	1.9	0.91	17.1

Glycine leaching – focus on copper



Effect of solids (%) on

(a) the extraction of Cu and
(b) Cu extracted and lost, and
(c) photos of liquid samples, slurry, filter cake and precipitates and
(d) UV–Visible spectroscopy analysis of the solution of cupric glycinate and the precipitates

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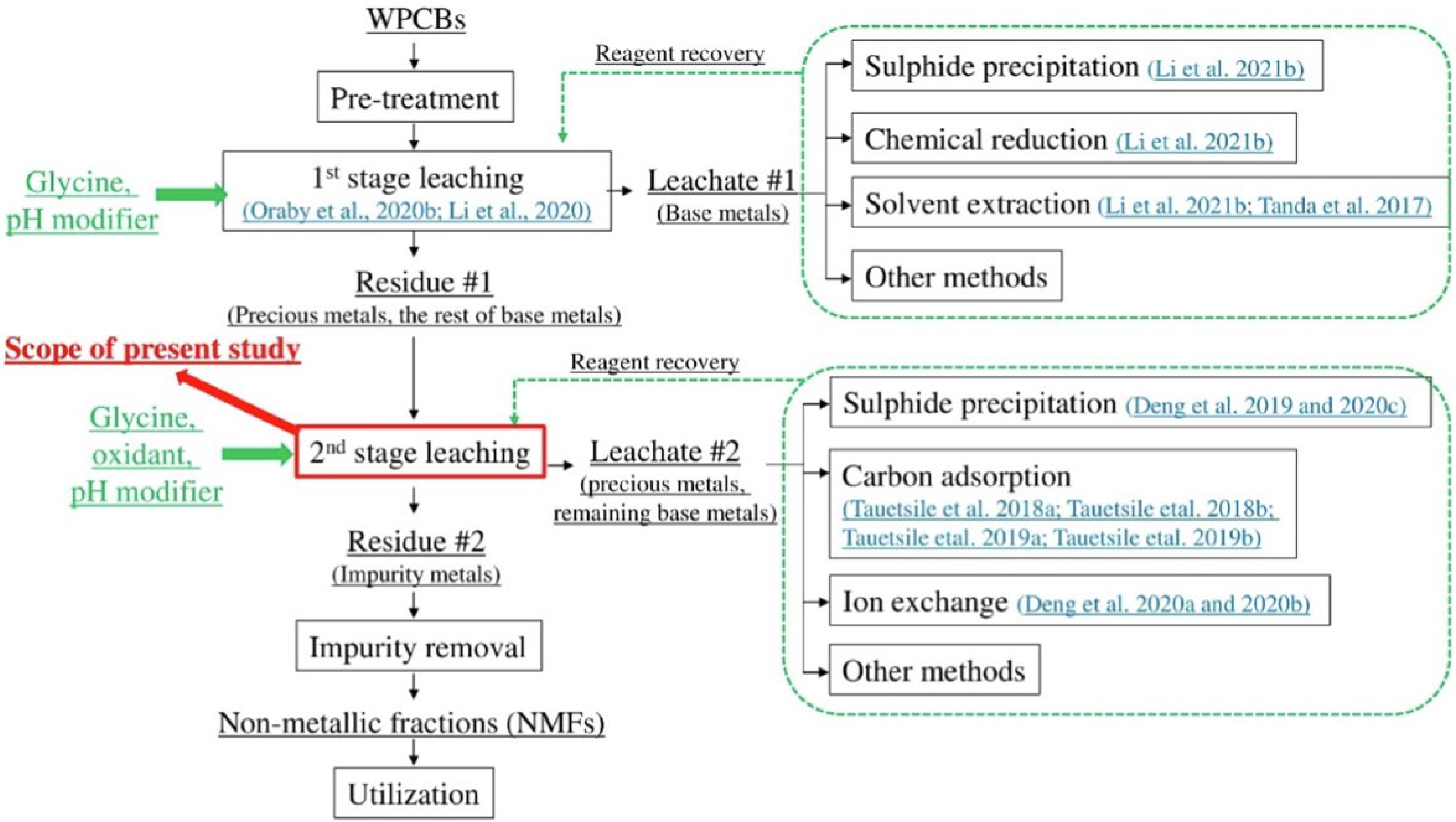
From base to precious metals extraction



The reactions with chemical oxidants for precious smetals extraction with glycine in cyanide-free environments

Permanganate can be used as an oxidant, but it also leads to partial glycine destruction:

$2MnO_4^- + 2NH_2CH_2COO^- + 0.5O_2\ (g) \rightarrow 2MnO_2\ (s) + 2C_2O_4^{2-} + 2NH_3\ (aq) + H_2O$, ΔG^o (25 °C) = − 1058.9 kJ/mol (R1)

$2MnO_4^- + 3NH_2CH_2COO^- + 4H_2O \rightarrow 2MnO_2\ (s) + 3HCHO + 3NH_3\ (aq) + 3CO_2\ (g) + 5OH^-$, ΔG^o (25 °C) = − 534.5 kJ/mol (R2)

$3HCHO + 2MnO_4^- + OH^- \rightarrow 2MnO_2\ (s) + 3HCOO^- + 2H_2O$, ΔG^o (25 °C) = − 1092.8 kJ/mol (R3)

$3HCOO^- + 2MnO_4^- + H_2O \rightarrow 3CO_2\ (g) + 2MnO_2\ (s) + 5OH^-$, ΔG^o (25 °C) = − 729.0 kJ/mol (R4)

$HCOO^- + 2MnO_4^- + OH^- \rightarrow CO_2\ (g) + 2MnO_4^{2-} + H_2O$, ΔG^o (25 °C) = − 250.3 kJ/mol (R5)

$2Cu + O_2\ (aq) + 4Gly^- + 2H_2O \rightarrow 2Cu(Gly)_2 + 4OH^-$, ΔG^o (25 °C) = − 219.0 kJ/mol (R6)

Anode reaction:

$Au + 2Gly^- \rightarrow Au(Gly_2)^- + e^-$, $E^o = 0.63$ V (R7)

Cathode reactions:

O_2 (from ambient air) $+ 2H_2O + 4e^- \rightarrow 4OH^-$, $E^o = 0.40$ V (R8)

$MnO_4^- + 2H_2O + 3e^- \rightarrow MnO_2\ (s) + 4OH^-$, $E^o = 0.60$ V (R9)

$Cu(Gly)_2 + e^- \rightarrow Cu(Gly)_2^-$ (R10)

Overall reactions:

$2Au + 4Gly^- + 1/2O_2\ (aq) + H_2O \rightarrow 2Au(Gly_2)^- + 2OH^-$, ΔG^o (25 °C) = 187.6 kJ/mol (oxygen as oxidant) (R11)

$3Au + 6Gly^- + MnO_4^- + 2H_2O \rightarrow 3Au(Gly_2)^- + MnO_2\ (s) + 4OH^-$, ΔG^o (25 °C) = 229.9 kJ/mol (permanganate as oxidant) (R12)

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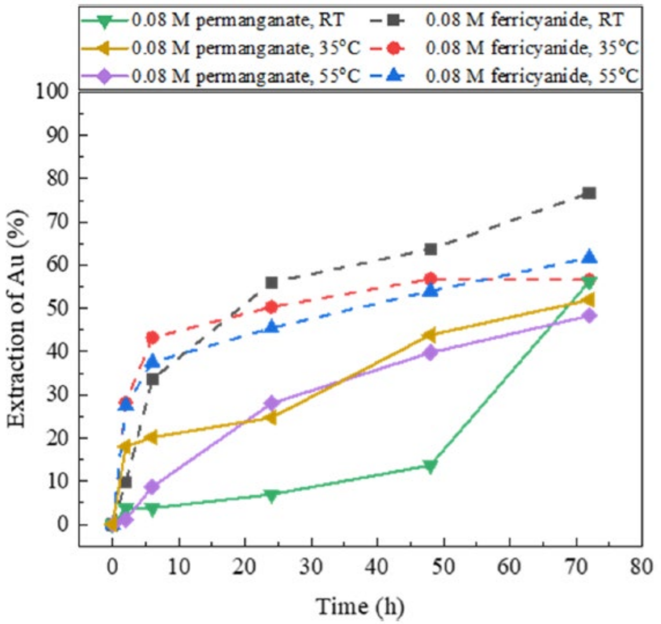
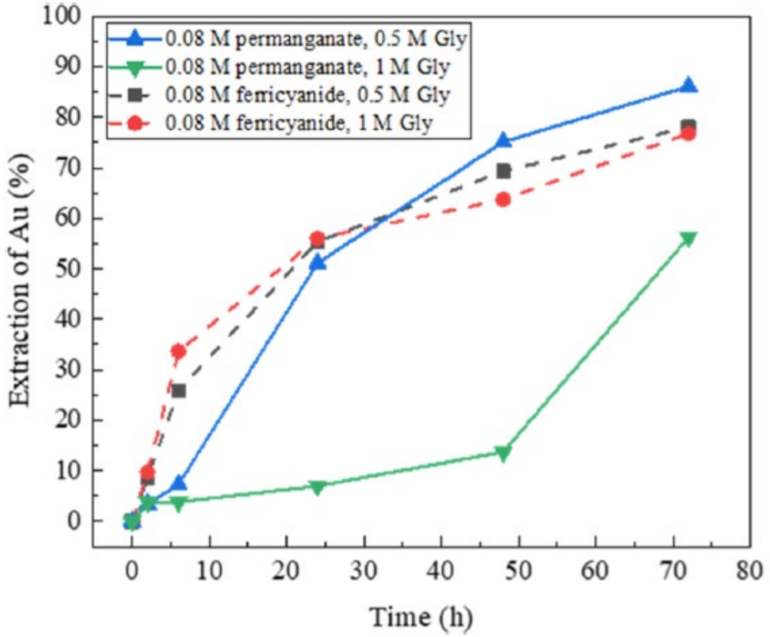
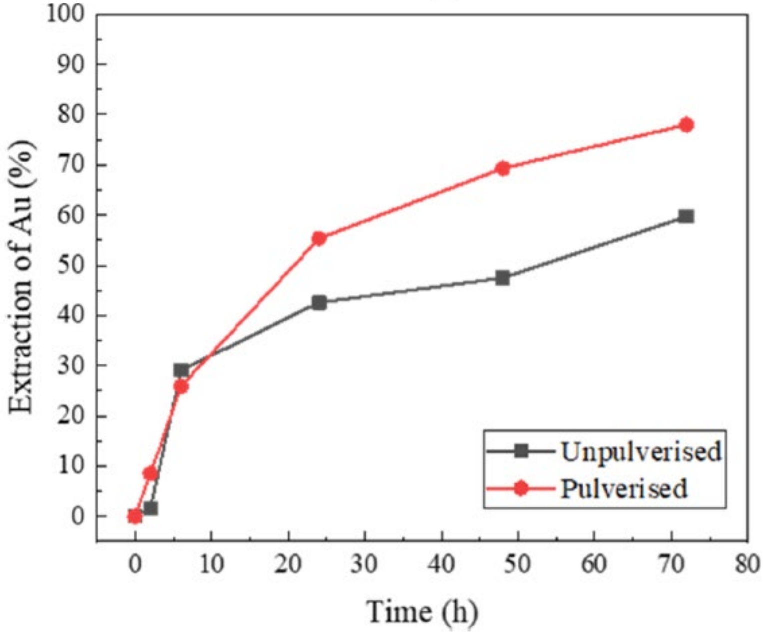
The role of various reaction parameters on gold dissolution



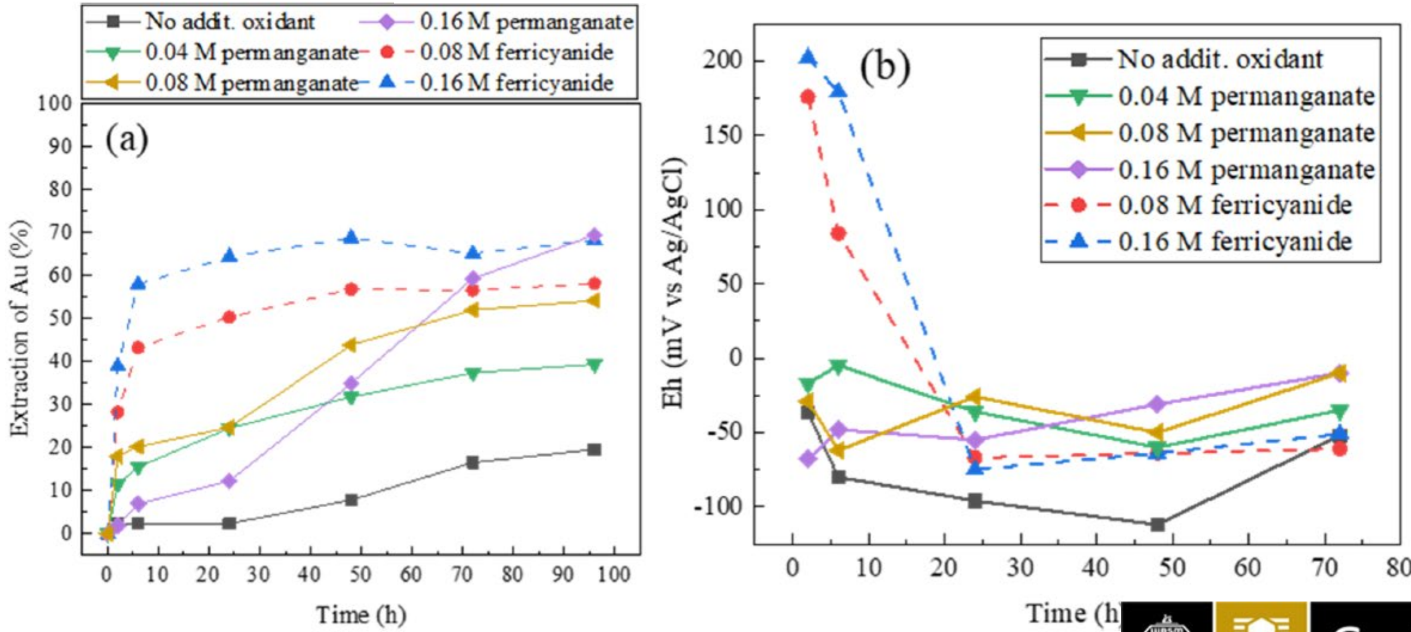
Effect of oxidant type and temperature



Effect of oxidant type glycine concentration



Effect of state of grinding



Effect of oxidant type and concentration

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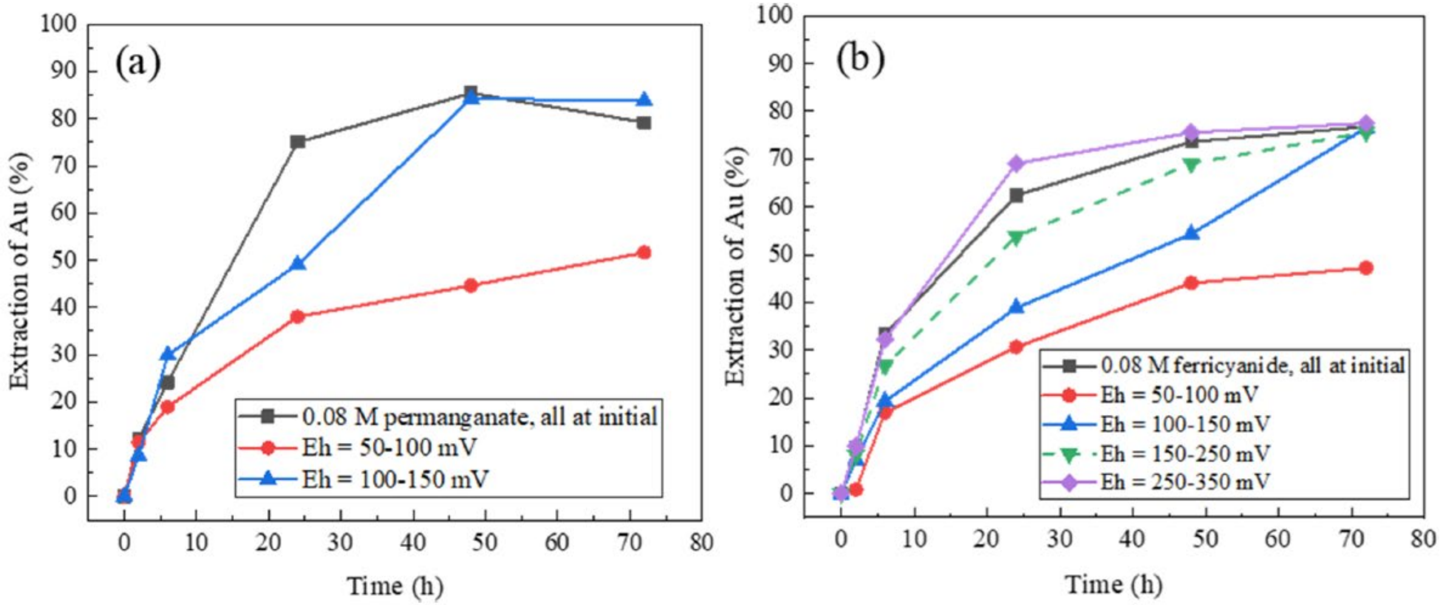
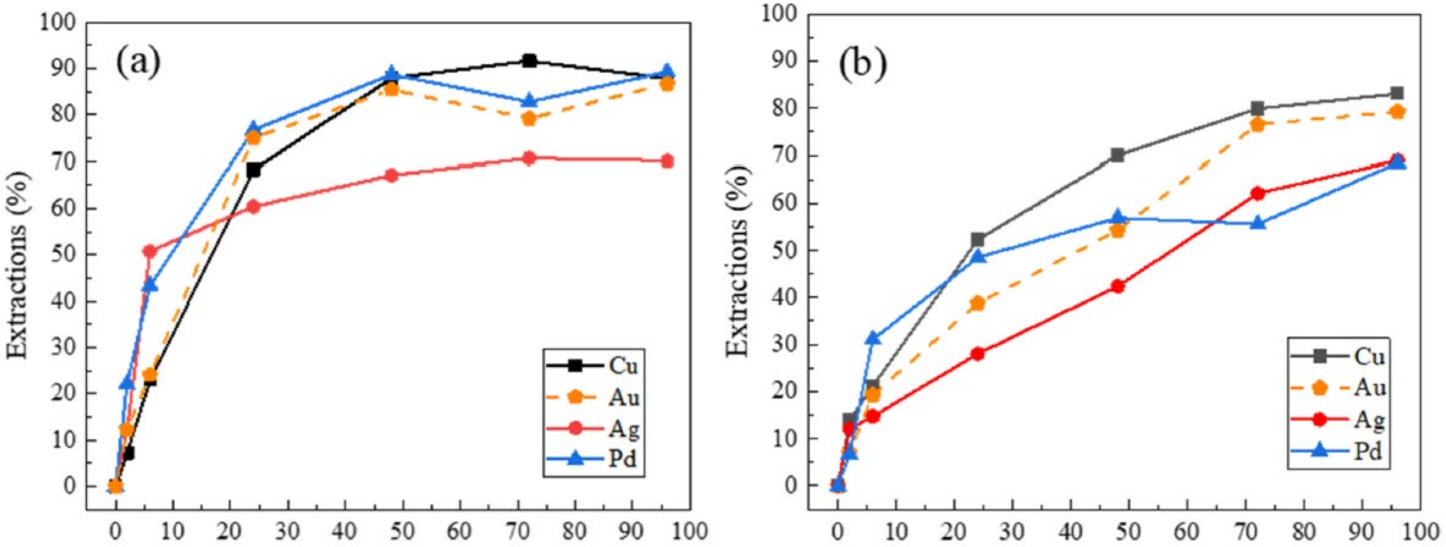
The role of various reaction parameters on precious metal dissolution



Effect of Eh (mV vs Ag/AgCl), controlled by adding

(a) permanganate and
(b) ferricyanide,

on the extraction of gold (0.5 M glycine, RT, pH 11).



Extraction of major metals under the recommended conditions of 0.5 M glycine, RT, pH 11, 2% solids,

(a) 0.08 M permanganate and
(b) Eh controlled by ferricyanide at 100–150 mV

Extraction of precious and base metals in this (96 h) and previous studies using the same batch of WPCBs sample

	Precious metals			Base metals							
	Au	Ag	Pd	Cu	Zn	Pb	Al	Ni	Co	Fe	Sn
Gly-KMO [a]	86.8	70.2	89.3	87.9	17.5	0.1	5.1	4.3	13.3	0.03	1.6
Gly-KFC [b]	79.3	69.0	68.5	83.1	7.2	38.6	5.7	1.8	–	N/A	1.3
CSG [c]	90.1	89.4	70.1	81.0	15.0	1.9	8.3	2.9	–	–	0.3
Intensive CN [d]	91.1	89.2	–	93.6	–	–	–	–	–	–	–

[a] Gly-KMO: glycine-permanganate leaching in this study;

[b] Gly-KFC: glycine-ferricyanide leaching in this study;

[c] CSG: cyanide-starved glycine leaching from Li et al. (2021a);

[d] intensive CN: intensive traditional cyanidation with 3.5 g/L cyanide in two duplicated stages from Li et al. (2021a); "-" denotes below detection limit or not reported; "N/A" denotes not applicable due to the interference from ferricyanide addition.

Conclusions

- The dominant base and precious metals in crushed waste printed circuit boards can be effectively leached from their matrices without thermal treatment, strong acids, high temperatures or high pressures or preconcentration of the metals
- Strong oxidizing acids, such as HNO3, aggressive corrosive acids such as HCl, or volatile and toxic alkaline reagents such as NaCN or concentrated ammonia can be averted
- Metal recovery from solution has been demonstrated in numerous published research articles
- Leach times are typically long (48 – 72 hours) given the mild conditions used
- Removal of epoxy and other polymer coatings (through say thermal treatment) is expected to further benefit metal removal and recovery
- The mildness of the process conditions and allows the building of multiple modular plants where even smaller towns can preprocess their e-waste to mixed metal precipitates (which can be sent to a refiner) and a plastic substrate residue which can be incinerated
- All the metal recovery happens under mild alkaline conditions (pH 8.5 – 11.5) and atmospheric pressure and mild temperatures (<60 °C)
- Certain amino acids are better for certain metals than others and conditions need to be optimised for a particular system

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Thank you!

Prof Jacques Eksteen

Jacques.Eksteen@curtin.edu.au

WA School of Mines, Minerals, Energy & Chemical Engineering

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