Journal of Undergraduate Chemistry Research, **2025**, 24 (3), 54

MECHANOCHEMICAL CHELATION AND GREEN EXTRACTION OF METALS FROM WASTE MOBILE PHONES

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Abstract

This study presents the use of mechanochemical chelation of metals from waste mobile phones as a green method of extracting critical and economic constituents from pulverized samples. Whole mobile phones, minus the battery, were mechanically pulverized and separated by particle size. The resulting powder was added, along with an organic chelating agent (citric acid, ethylenediamine tetraacetic acid, or sodium thiosulfate) and water, to a mill. The slurry was milled, separated, and both solid and liquid fractions analyzed for by Energy-Dispersive X-ray Spectroscopy and X-Ray Fluorescence Spectroscopy to identify elements extracted into solution. This method was successful in extracting 19 total elements, with specificity differences between chelate types.

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Keywords: Mechanochemistry, Metal-Ion Chelation, E-Waste, WEEE, Green Chemistry

Received: June 11, 2025 Accepted: June 27, 2025 Revision received: June 30, 2025 Published: July 1, 2025

Introduction

With the pace of modern technology accelerating and the rise of global participation in digital communities, there is an increase in the number of raw resources being used in personal electronic devices. In turn, this also means that due to the short duration of the typical use of modern electronic technology, there is an increase in the number of devices entering the waste stream. It is estimated that there was 52.2 million metric tons of Waste Electrical and Electronic Equipment (WEEE) in 2021, with a projected increase to 74.7 million metric tons by 2030.[1,2] This represents an approximate 40% increase within the next decade and underscores the need for better end-of-life options for electronic waste (e-waste). Virgin materials are finite, and extraction often has negative environmental and human health impacts.[1] Paired with the 2020 global report that only 17.4% of e-waste was properly collected and recycled, this tells us there is a need for improvements to the recycling systems we currently utilize.[3] Currently, only 1% of the world's rare earth elements are recycled.[4]

Current physical recycling technologies implement a variety of increasingly effective methods to collect, sort, wash, and grind materials for mechanical processing, but due to the complexity of the WEEE stream, many of the categories of waste require manual disassembly to implement recycling methods.[5] Kohl et al. published that with exception to the screens, approximately 81.96% - 96.66% of desktop computer waste can be reclaimed, and smaller devices are reported to share in high potential yields.[1,4] Individual components such as printed circuit boards (PCBs) and small IT and telecommunication devices, such as mobile and smartphones, represent 3-7% and 8.8% of WEEE mass, respectively.[1,2] PCBs contain up to 60 elements while phones contain up to 40, which for PCBs that were in larger components, represents 80% of the recyclable value in 1% of the mass.[2,3]

The Basel Action Network has been monitoring toxic exportation since the 1970s and in conjunction with The World Bank Group detail problematic methods abroad of "recycling" WEEE that include open pit burning, inadequate storage of potentially dangerous components, open air landfills, and unsafe aqua regia use and disposal, all leading to the liberation of toxins into the air, waterways, and soil around the materials.[6,7]

Mechanochemical extraction is increasingly being used in WEEE reclamation that is evident by the number and quality of studies being reported.[8] There is a growing need for processes that allow for a greener separation of toxic constituents that are being abandoned after industrial use leading to discarded hazardous wastes containing economically important elements that contribute to negative health outcomes.[9] Mechanochemical processes are being used to recover Co and Li from batteries and lead glass, to determine mobile phone compositions and to conduct toxicological impact studies.[2,10,11] Oxide synthesis is being investigated by milling with sulfur and various chelates to reduce the amount of pollution and energy consumption in traditional sulfidization reactions with zinc, nonferrous metal oxides, and lead oxides.[9,12,13]

This study investigates whether a ball mill, with liquid assisted grinding (LAG), is capable of extracting multiple critical and economic constituents from pulverized WEEE samples using economically viable chelates. Extraction of multiple elements simultaneously in this system could represent a method of mass capture that would render WEEE safer for disposal and could offer potential mechanisms for the separation of problematic constituents in a more environmentally sustainable way.

Experimental Methods

Materials – Research grade anhydrous citric acid ($C_6H_8O_7$), ethylenediamine tetraacetic acid tetrasodium salt hydrate ($Na_2EDTA*xH_2O$, EDTA), and sodium thiosulfate heptahydrate ($Na_2S_2O_3*7H_2O$), were procured from commercial sources and used without further purification. Two mobile phones, a Samsung SM-G530T UD and a Motorola MB525 (Device 1 and Device 2, respectively), were pulverized to create a solid powdered sample (see Sample Preparation below).

Instrumentation – All liquid-assisted grinding (LAG) syntheses were performed on a Retsch Mixer Mill MM400 using a 10-mL stainless steel milling jar and 5 mm stainless steel balls. Fourier transform infrared/attenuated total reflectance (FTIR/ATR) spectroscopy was performed on a Nicolet iS50. 40 scans, with background correction between each sample was performed on each sample. Elemental analysis of solid samples was performed on a Quanta 250 Environmental Scanning Electron Microscope (SEM) equipped with Energy Dispersive X-ray analysis (EDX). Elemental analysis of liquid samples was performed using a Rigaku

Supermini 200 Wavelength Dispersive X-ray Fluorescence Spectrometer (XRF).

Sample Preparation – Device 1 and Device 2 were individually ground in a BlendTec Designer 635 Blender in a hood for 45 seconds at 2/3 power. The largest pieces were then manually cut with tin snips into smaller pieces and the whole sample was ground again for 45 seconds at 2/3 power. Care was taken with handling the electrostatically attractive powders, which were produced in aundance with this pulverization method. After grinding, the particles were fractionally separated using a five-piece W.S. Tyler Company US Standard Sieve series to ASTM specifications, with the smallest sieve (Sieve #120) separating particles below 125 µm. Approximately 1.25 g of the pulverized solid sample with a grain size of < 125 µm was combined with a dilute nitric acid solution (0.01 M) and the selected chelate for the specific milling to a 10 mL stainless steel grinding jar with two 5 mm stainless steel balls and milled. The resulting milled slurries were filtered by Hirsch funnel and washed with distilled water to separate the liquid and solid fractions. A blank was setup using 1.25 g of pulverized solid sample with only dilute nitric acid solution to distinguish between soluble metals versus chelated metals.

Extraction with Citric Acid – Powdered material of Device 1 or Device 2 (1.25 g), citric acid (1.00 g, 5.20 mmol), and 2.1 mL dilute nitric acid solution were added to the stainless-steel grinding jar with two stainless steel balls and ground as described above, then separated using a Hirsch funnel. Product slurry of Device 1 resulted in a slightly cloudy solution with a white fluffy suspended solid. The dried solid fraction appeared to have 2 layers, with the bottom 2/3 layer being a lighter, dull, metallic gray, and the top 1/3 layer being a stark white. Product slurry of Device 2 resulted in a transparent, slightly yellow solution. The dried solid fraction appeared to have 2 layers; the bottom 1/2 was white with black spots, and the top 1/2 was a dull gray color with metallic spots on the surface.

Extraction with EDTA – Powdered material of Device 1 or Device 2 (1.25 g), EDTA (2.00 g, 4.60 mmol), and 2.1 mL dilute nitric acid solution were added to the stainless-steel grinding jar with two stainless steel balls and ground as described above, then separated using a Hirsch funnel. Product slurry of Device 1 resulted in a slightly cloudy, green colored solution. The dried solid fraction had a thin top layer that was a dull gray with metallic spots that flaked off a white bottom layer. Product slurry of Device 2 resulted in a slightly cloudy copper-colored solution with a fine metallic-suspended solid. The dried solid fraction was white.

Extraction with thiosulfate – Powdered material of Device 1 (1.25 g), sodium thiosulfate heptahydrate (1.25 g, 5.04 mmol), and 2.1 mL dilute nitric acid solution were added to the stainless-steel grinding jar with two stainless steel balls and ground as described above, then separated using a Hirsch funnel. Product slurry of Device 1 resulted in a clear solution. The dried solid fraction was a fine gray solid.

Solid Elemental Analysis by EDX – Five replicate scans were completed of different areas of the solid samples before and after milling to obtain average weight % and average atomic %. Signal differences due to the blank sample were subtracted out from the chelated analyses.

Liquid Elemental Analysis by XRF – Analysis was performed under the MicroFilter Qualitative condition and scanned for the preset "Heavy" (including elements F through U). Additionally, Ca, K, S, Si, Al, Mg, Na, and F were individually selected for scanning. Peak identification and qualitative analysis were collected for each sample. Signal analyses from the blank were subtracted from the chelated analyses.

Analysis of chelate by FTIR/ATR – Analysis was performed for the citric acid and EDTA extracts and the signal for the carbonyl stretch of free acid ($1637\ cm^{-1}$ – $1776\ cm^{-1}$) was qualitatively compared to the signal of the metal chelate ($1518\ cm^{-1}$ – $1637\ cm^{-1}$) according to the published method to show that the chelate is chemically interacting with the metals.[14]

Results and Discussion

The processing of the samples is summarized in Figure 1. This study used a blender to pulverize the sample, and while this was highly effective at achieving very fine particles to work with for analysis, it was not sufficient in pulverizing the entire phone. The method created excess airborne electrostatically attractive powders. In future experiments, a different method will need to be explored which yields less airborne powders. Each phone had pieces that were significantly large and intact. The fraction with the smallest particle size was only 3% of the total mass for Device 1 and 1% of the total mass for Device 2. Care was taken in this work to prioritize components that would contain extractable metals, including circuit boards, and any electronics. For this method to work, a larger portion of the total mass must be further reduced in size so that there is more of a homogeneous composition throughout. Because some degree of manual disassembly is widely reported to be a part of processing the electronic waste, a novel method of treating the entire device to reduce processing time may still increase the likelihood of industrial application.

EDX analysis of the pre-milled powdered devices detected the presence of sixteen elements. Blank milling was performed on both devices to find which elements were soluble in only dilute nitric acid solutions. The remaining elements that were extracted into solution with the addition of ligands are listed in Tables 1 and 2. The largest contributor to the composition of the devices was silicon, which was not extracted during our process. The next largest set for Device 1 included aluminum and iron, accounting for 2.60% of the total mass. The remaining nine elements contributed 5.27% of the mass. For Device 2, the second largest set contained

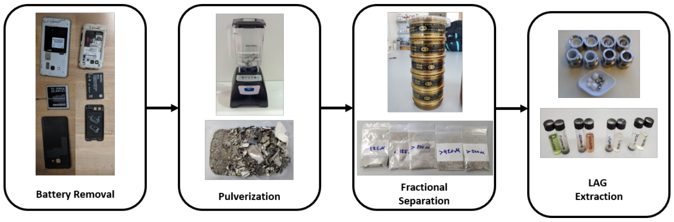

Figure 1. Schematic image showing (from left to right) the processing of Device 1 and 2. First, the battery was removed from both phones. Then they were individually pulverized in the blender leading to the ground particles. Next the produced powders were fractionally separated by size. Finally, the smallest powder size was used for LAG extraction of metals. Results are shown for each step for Device 1.

aluminum nickel and zinc, accounting for 4.75% of the total mass. The remaining eight elements contributed to 4.53% of the mass. EDX analysis of the post-milled solid fraction of Device 1 with citric acid and EDTA showed a reduction in concentration for six elements and five elements, respectively (see Table 1). EDX analysis of the post-milled solid fraction of Device 2 with citric acid and EDTA similarly showed a reduction in concentration for seven elements and five elements, respectively (see Table 1). In general, the citric acid extracts showed a higher % extractions than EDTA for all comparable elements in Device 1 and only had slightly less extraction of copper in Device 2. This could be due to the higher mole amount of citric acid in our samples compared to EDTA. Beyond this analysis, XRF analysis of the post-milled liquid-fraction citric acid and EDTA extracted Device 1 and 2 showed the presence of three and four additional elements extracted, respectively (indicated with double asterisks). These other elements were in low enough concentration that they were not originally detected by EDX, but their presence in the post-milled liquid-fractions of the ligands above the blank was evidence that they indeed came from the devices and were successfully extracted. There were slight differences in the identity of extracted elements between citric acid and EDTA with citric acid solely able to extract titanium, chromium, tin, and neodymium from both devices while EDTA had more of an affinity to copper and gallium in both devices. While both ligands utilize carboxylic acid functional groups for coordination to metal ions, citric acid is multidentate capable of forming up to three coordination bonds in a metal-ligand complex. The structure and size of this ligand allows it to have many conformations and may form mononuclear and binuclear complexes. EDTA is also multidentate, and can bond up to six times, if it also uses its amine groups for coordination.

In order to investigate the effect of a softer base for LAG extraction, powdered sample of Device 1 was milled with sodium thiosulfate. The results of this milling are summarized in Table 2. EDX analysis of the post-milled solid-fraction showed a reduction in concentration of eight elements. XRF analysis of the post-milled liquid-fractions revealed the presence of three additional elements extracted from Device 1. In general, thiosulfate appeared to have a higher preference for heavier atoms including second row transition elements molybdenum, ruthenium, and silver over first row transition elements like chromium, iron, nickel, and copper. Additionally, thiosulfate was able to successfully extract these heavier transition metals, whereas citric acid and EDTA were not, which could be attributed to the hard/soft acid/base interactions where sulfur would have a higher affinity to larger, softer metal cations. Qualitative extraction results are summarized in Figures 2 and 3.

To provide further evidence that the ligands are fully bonding with the metal cations extracted from the powdered samples, FTIR was performed on the liquid-fraction of the citric acid and EDTA extracts (Figure 4), in which the carbonyl stretch of the bonded carboxylates are compared to the same stretch of the free acid.

Table 1. Comparative Extraction Results from Citric Acid and EDTA Ligands with Device 1 and 2

Element	Device 1					Device 2				
	Init. Conc.*	Citric Acid		EDTA		Init. Conc.*	Citric Acid		EDTA	
		Final Conc.*	% Ext.	Final Conc.	% Ext.		Final Conc.*	% Ext.	Final Conc.*	% Ext.
Al†‡	70500	30104	57.3	67328	4.5	12580	2944	76.6	10290	18.2
Ti	2460	1112	54.8			4460	3595	19.4		
Cr†	2700	1723	36.2			2860	2508	12.3		
Mn†						**	**	N/A		
Fe†‡	15100	1720	88.6	13110	13.2	1325	60	95.4	379	71.4
Ni†‡	4240	2497	41.1	2854	32.7	20620	16805	18.5	18640	9.6
Cu†‡	7733			2548	75.2	4940	3868	21.7	3675	25.6
Zn†	5100	204	96.0	795	84.4	11775	5935	49.6	8384	28.8
Ga	**			**	N/A	**	**	N/A	**	N/A
Sn	**	**	N/A			**	**	N/A		
Nd	**	**	N/A			**	**	N/A		

* Concentrations (ppm) calculated from EDX data of solid samples pre and post milling. Elements and concentrations obtained from blank sample were subtracted from data.

** Elements qualitatively detected by XRF analysis in liquid extract, but not by EDX analysis of solid samples. Limitations of EDX explained in discussion section.

†Toxic elements[1]

‡Economic elements[1]

Table 2. Extraction Results from Sodium Thiosulfate Ligand with Device 1

Element	Init. Conc.*	Final Conc.*	% Ext.
Cr†	2700	2603	3.6
Mn†	**	**	N/A
Fe†‡	15100	5829	61.4
Ni†‡	4240	530	87.5
Cu†‡	7733	2165	72.0
Zn†	5100	347	93.2
Mo	7860	354	95.5
Ru	3833	203	94.7
Ag†‡	1800	72	96.0
Sn	**	**	N/A
Nd	**	**	N/A

* Concentrations (ppm) calculated from EDX data of solid samples pre and post milling. Elements and concentrations obtained from blank sample were subtracted from data.

** Elements qualitatively detected by XRF analysis in liquid extract, but not by EDX analysis of solid samples. Limitations of EDX explained in discussion section.

†Toxic elements[1]

‡Economic elements[1]

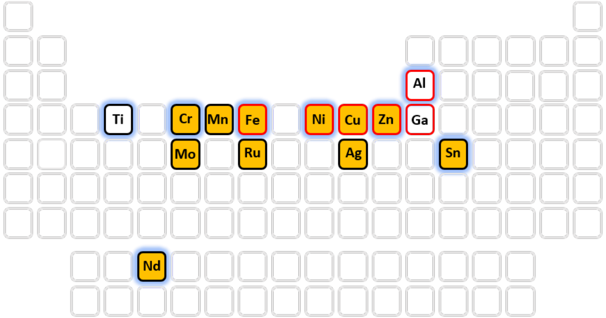

Figure 2. Periodic Map of Extraction Results of Device 1. Image of the elements successfully extracted through LAG mechanism of powder from Device 1. Data obtained either from quantitative data from EDX analysis, or qualitative date from XRF analysis. Extraction from citric acid is shown by a shadow around the element. Extraction with EDTA is shown by a red border around the element. Extraction with thiosulfate is shown by an orange background in the element.

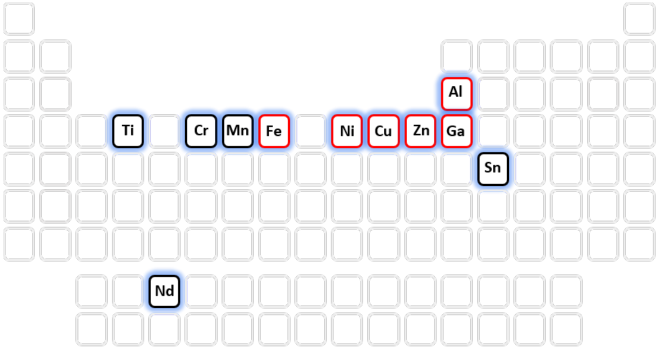

Figure 3. Periodic Map of Extraction Results of Device 2. Image of the elements successfully extracted through LAG mechanism of powder from Device 2. Data obtained either from quantitative data from EDX analysis, or qualitative date from XRF analysis. Extraction from citric acid is shown by a shadow around the element. Extraction wih EDTA is shown by a red border around the element.

There is a clear shift to lower wavenumbers, indicating the weakened carbonyl bond due to its interaction with the metal, consistent with the literature.[14]

Sodium thiosulfate and EDTA are both recognized by the USEAP as useful ligands in reducing the toxicity of freshwater environments containing certain elemental pollutants.[15] The toxicity of mobile phone constituents has been investigated using the USEPA's method 1311 to simulate the conditions present in landfills, revealing the most significant human carcinogenetic and ecotoxicity risks are associated with copper and chromium, both being present in the liquid fraction.[2]

The ligands selected for this study all share the ability to be recycled after separation under relatively simple conditions. This increases the sustainability of the process when compared to solvent methods.[16] This comes with its downsides; the resulting leachate is a very complicated matrix and additional study would be required to determine if individual elements can be environmentally and economically separated further.

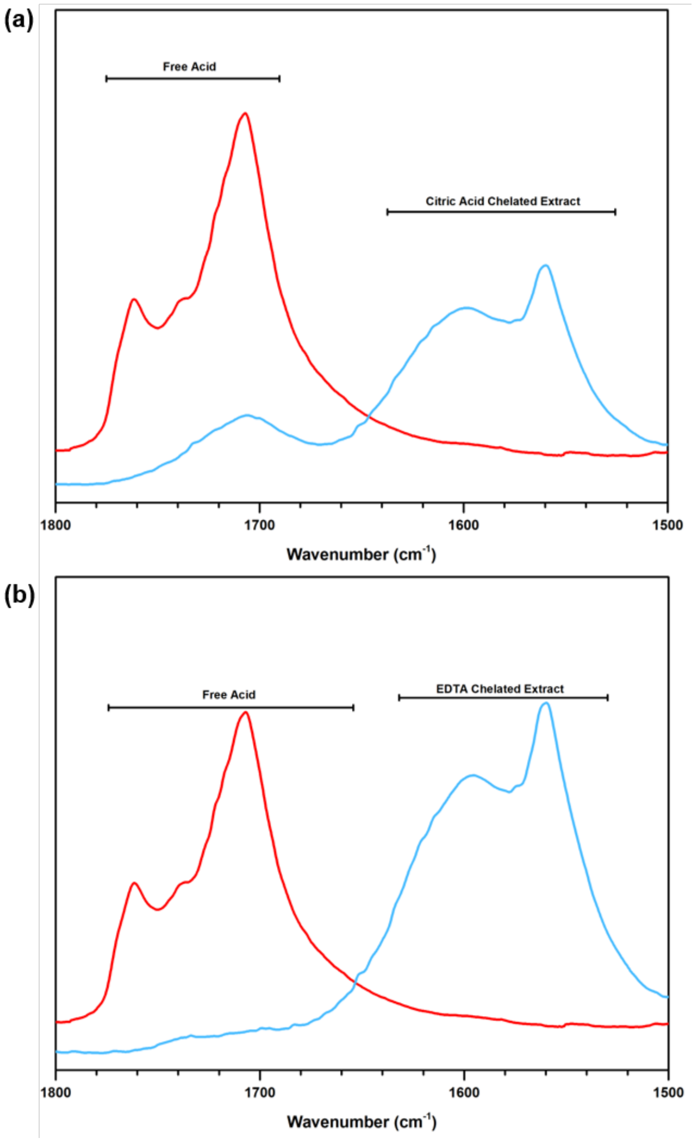

Figure 4. Overlaid FTIR spectra of Chelate Extracts of Device 1.FTIR spectra results of the chelate LAG reaction between powder of Device 1 with (a) Citric Acid and (b) EDTA compared to the pure ligands.

Conclusions.

In this experimental study we investigated a novel method for sample pulverization, the effect of three ligands during mechanochemical LAG extraction of samples, and the mass extraction of elements from WEEE. Pulverization by blender alone was sufficient in producing a powdered sample with small enough particles for accurate analysis, however it left multiple fragments that were large enough to prevent analysis of the entire phone sample. Citric acid extracted the most elements in the highest relative concentrations, second to thiosulfate, with EDTA extracting the least number of elements. The experiments demonstrate ligand selectivity and viability, but not optimized recovery. To achieve a fully quantitative analysis of the system, further elemental analysis is being explored utilizing inductively coupled plasma-optical emission spectroscopy (ICP-OES).

Acknowledgment.

The authors acknowledge the financial support from Weber State University's Office of Undergraduate Research. We would like to thank Amanda Gentry and David Matty for help with XRF, Colin Ingelfield and Michelle Arnold for help with EDX, and Leigh Komperda for instrumentation help.

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