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Selective recovery of gold and silver from electronic wastes through a sequential process of Qalkari and room-temperature hydrometallurgy

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Abstract

This work was focused on the selective recovery of gold and silver from electronic wastes using a sequential process of pyrometallurgy (Qalkari) and room-temperature hydrometallurgy. In the first step, electronic wastes underwent Qalkari recycling, yielding tablets containing precious elements (Qalkari furnace product) and melting slag (Qalkari furnace waste). In the subsequent hydrometallurgy phase, the nitric acid concentration and the input solid amount were optimized for the effective room-temperature recovery of gold. Due to the successful separation of precision elements and disturbing substances in Qalkari, the gold recovery efficiency of 99.99% was obtained at the acid concentration of 50% (v/v) and the solid input of 15% (w/v). Afterwards, HCl, NH_4Cl, and NaCl were used for silver recovery from the Qalkari-processed gold-recovered leaching solution, leading to the efficiency of 99.99%. But NH_4Cl was recognized as the most effective precipitant as it promises the most enhanced potential for the possible subsequent recovery of palladium. In conclusion, this study draws the effectiveness of Qalkari in recycling electronic wastes, with a significant impact on the

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efficiency of succeeding room-temperature hydrometallurgical processes for gold and silver recovery within a reasonable leaching time.

Keywords: E-wastes; Cupellation; Sedimentation; Industrial development; Dissolution

1. Introduction

In recent years, electronic wastes have grown the fastest among various single wastes [1]. The European continent has particularly excelled in documenting and managing 42.5% of these wastes [2]. In general, the successful implementation of waste management strategies is contingent upon the existence of industrial methods for recovering materials from wastes. While significant progress has been made in recent years, many of these methods are not yet industrialized [3, 4], and some are restricted due to environmental regulations [5, 6]. Consequently, the development of appropriate methods that mitigate pollution remains crucial.

The presence of diverse polymeric materials [7-9] and over 57 elements [2, 10, 11] in electronic wastes necessitates their initial purification before hydrometallurgy. Also, the environmental effects and management of liquid wastes are more complicated than those of solid wastes [12], provided that hydrometallurgy is originally used in recycling electronic wastes. For this purpose, the industry often resorts to pyrometallurgical methods such as Noranda, Boliden Rönnskär Smelter, and IsaSmelt Umicore [13-15]. However, it is worth considering that these methods involve specialized equipment, and complexity does not necessarily imply absolute functional superiority. There are other more straightforward industrial processes, for instance, Qalkari that has been originally used in Iran and worthy of further attention. Indeed, Qalkari represents an updated version of conventional cupellation to separate precious elements in terms of Qalkari furnace product from Qalkari furnace waste consisting of some disturbing materials. Qalkari offers the advantage of simultaneous operation on various electronic wastes, and its filtered dusts can be used for the recovery of elements like selenium. The Qalkari furnace waste can be also reused for melting subsequent electronic wastes, particularly lead monoxide (litharge) as an oxidant for collecting disturbing elements. Despite these benefits, there is a lack of published scientific knowledge on the analysis of the Qalkari furnace waste, Qalkari furnace product, and the efficiency of subsequent hydrometallurgical stages.

After the initial pyrometallurgical recycling of wastes, nitric acid is typically used for the hydrometallurgical recovery of gold. This is because it provides a one-step process for both leaching of other elements and the precipitant-free recovery of gold. The temperature of this hydrometallurgical process is a significant factor influencing the initiation and operation costs of the plant, as higher temperatures necessitate more complex equipment or maintenance due to the increased need for the inspection, repair, and shutdown of the plant. Despite this fact, the recovery of gold through the one-step process of using nitric acid at temperatures close to room temperature has not been well developed, primarily due to the limited success observed in the few attempts made. The leaching level of other elements is used as the criterion of success in this context due to the selective character of nitric acid that leaves certain elements like gold undissolved, while dissolving others. Typically, Bas et al. [16] investigated the impact of parameters like temperature (30-70 °C), time (≤120 min), concentration (1-5 M), and solid ratio (2-10 %) on leaching of Ag and Cu. At 30 °C along with the optimal set of the other variables, a dissolution efficiency of only 15% was achieved for copper. This parameter for silver and copper reached 68 and 99.9%, respectively, during leaching at 70 °C for 120 min. Also, Kumar et al. [17] focused on leaching of Cu, Ca, Pb, Al, and Fe under various temperature (35-90 °C), time (≤300 min), concentration (1-4 M), solid ratio (2.5-12.5 %), and agitation speed (400-800 rpm) conditions. With the optimal set of the other variables, the best results were obtained at 90 °C with the leaching efficiency of 96.0, 25.4, 88.1, 33.7, and 59.1% for Cu, Ca, Pb, Al, and Fe, respectively. These values at 35 °C with its own optimal set for the other variables were insignificant. Rao et al. [18] also recovered gold in a two-step process, including leaching in nitric acid at 30°C and then the separation of gold via a solvent extraction method using sodium bromide at 70 °C. Despite the suitability of the leaching efficiency for some elements, a low leaching efficiency of 30.8 and 8.0% was reported for Sn and Ag, respectively.

For silver recovery, chlorine-based precipitants are used due to their cost-effectiveness and ease of operation. Several studies have been focused on optimizing other variables while using a fixed chlorine-based precipitant. Typically, the silver recovery efficiency of 87.3 and 98% has been reported using NaCl [19] and HCl [20], respectively. However, Panda et al. [21] compared various chlorine-based precipitants for silver recovery from a nitric acid solution. The efficiency of silver recovery using KCl, HCl, NaCl, $CuCl_2$, and $CaCl_2$ was reported to be very similar, quantified as 99.94, 99.72, 99.94, 99.95, and 99.96, respectively, while leaching had been previously conducted at 80 °C. The question remaining in this context is how various chlorine-based precipitants affect the sedimentation of other elements, particularly palladium, during silver recovery. This is significant as the recovery of this precision element is typically pursued after silver recovery during the recycling of electronic wastes. To address the scientific gaps described above, this research work was focused on the characterization of the Qalkari furnace waste and product, the one-step room-temperature recovery of gold from the Qalkari furnace product, and the comprehensive comparison of HCl, NH_4Cl, and NaCl precipitants for

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room-temperature sliver recovery from a Qalkari-processed gold-recovered leaching solution. One of the other special characteristics of this work, compared to the similar studies, is to analyze a wide range of elements apart from the elements targeted for recovery, a valuable database for the recovery of other elements.

2. Materials and methods

2.1. Materials

Electronic wastes utilized in this work included computer RAMs, mobile battery heads, wireless boards, SIM cards, gold-plated plugs (related to the aviation industry motherboard chips and surface capacitors, and telecommunication boards), and server parts. The total weight of scraps subjected to Qalkari treatment was 5 kg. Chemicals employed in the study comprised nitric acid (65%, Mojallali Industrial Chemical Complex), hydrochloric acid (37%, Mojallali Industrial Chemical Complex), hydrogen peroxide (35%, Mojallali Industrial Chemical Complex), sodium chloride (99.5%, Mojallali Industrial Chemical Complex), ammonium chloride (99.5%, Mojallali Industrial Chemical Complex), disodium tetraborate (borax 100%, Neutron Pharmachemical Co) in laboratory grades, and light sodium carbonate (Semnan Sodium Carbonate Company), lead ingot (Tadbir Sanat Ayandegan Qarn), and litharge (Qalkari furnace wastes from previous smelting) in industrial grades.

2.2. Qalkari

The Qalkari furnace used in this study is shown in Fig. 1, while it had a 30 kg capacity and operated on natural gas fuel. During the Qalkari process, precious elements were collected

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along with copper from the electronic wastes, resulting in the formation of a tablet-shaped concentrate of precious metals as the furnace product. Interfering elements were collected under an oxidation process with litharge and removed as the furnace wastes in multiple stages.

Fig. 1. Components of the Qalkari furnace: chamber under the furnace (A), chamber on the furnace (B), blower (C), forsonga (D), gas fuel inlet pipe (E), and furnace lifting jack (F).

The Qalkari process involved several stages, including mixing the electronic wastes with borax, sodium carbonate, litharge, lead, and copper. The furnace was then heated to 600 °C, and the charging materials entered the furnace at 1300 °C. After oxidizing interfering elements by blowing compressed air, remaining melting materials were removed by tilting the furnace, forming a tablet that was subsequently quenched in water (Fig. 2).

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Fig. 2. The process of preparing the Qalkari samples: waste entering the furnace (A), heating the furnace to enter the waste and melting aids (B), entering the waste into the furnace (C), entering the litharge and lead into the furnace (D), entering the melting aid into the furnace (E), melting the electronic wastes (F), stirring the melt to form an eddy current (G), Initial discharge of melting slag (H), final discharge of melting slag (I), lowering the temperature and forming a tablet after the final discharge of the melting slag (J), removing the tablet from the

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sphere (K), quenching the tablet in water (L), the product of the Qalkari furnace (814 g tablets) (M), and the waste of the Qalkari furnace (17 kg ingots) (N).

For inductively coupled plasma mass spectrometry (ICP-mass) testing specimen preparation, representative samples were chipped from different points of the Qalkari furnace product. For Qalkari furnace waste sampling, samples were taken from all layers and then powdered to appropriate dimensions using mechanical milling (Fig. 3).

Fig. 3. Preparing a sample from the product of the Qalkari furnace (A) and preparing a sample from the waste of the Qalkari furnace (B).

2.3. Hydrometallurgy

Industrial methods were employed for the recovery of precious elements (gold and silver) from the Qalkari furnace products, divided into G-1 and G-2 phases for gold and an S phase for silver. In the G-1 phase experiments, the primary goal was to measure the dissolution of elements in the shortest time at a ratio of 10% w/v (the ratio of the input solid to the solution) without altering the temperature. The independent variable in this phase was the acid concentration, while the dependent variable was the amount of the solid dissolution in the solution. The time parameter, based on the completion of dissolution in the strongest solution, was set at 30 min, with a stirring speed of 250 rpm to prevent the formation of air columns. Three samples, each weighing 10 g, were prepared from shavings of the Qalkari tablets. Subsequently, three solutions labeled A (100% nitric acid), B (50% nitric acid + 50% distilled water), and C (25% nitric acid + 75% distilled water), each with a volume of 100 ml, were prepared.

Experiments of the G-2 phase were conducted based on the most successful solution of the G-1 phase. Success was measured by the highest solid dissolution efficiency with the minimal acid usage. By eliminating heating and increasing the solution concentration, the economic aspects of the process are influenced, but this can be offset by increasing the solid loading into the solution. The time parameter in this phase was set at 1 h, based on the conclusion of interactions, color stabilization, and pH rise for all the three solutions. The independent variable in this experiment was the ratio of the solid to the solution, while the dependent variable was the amount of the solid dissolution in the solution. Three conditions, named G-2-1, G-2-2, and G-2-3, were considered 15, 20, and 40 g of the input solid, respectively, in the optimal G-1 solution.

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The primary objective of the S phase was to compare three chlorine-based precipitants—HCl, NH_4Cl, and NaCl—at the same molar amount of chlorine to recover silver from the selected nitric acid solution of the G-1 phase. The independent variable in this step was the type of precipitant, while the dependent variable was the amount of silver recovery from the solution. All the experiments in this phase were conducted at room temperature, and the time parameter was based on the conclusion of sedimentation and stabilization of the solution color within 30 min. For each sample, a 30 ml solution without dilution was prepared. 1 ml, 2.40 g, and 2.19 g of HCl, NH_4Cl, and NaCl, respectively, were used according to stoichiometric calculations.

2.4. Analytical techniques

The analyses employed in this study included scanning electron microscopy (SEM, PHILIPS XL 30), ICP-mass (Agilent 7500), X-ray fluorescence (XRF, PHILIPS PW 1410), X-ray diffraction (XRD, PHILIPS PW 1730), and peroxide fusion testing (utilizing inductively coupled plasma optical emission spectroscopy, ICP-OES, Agilent 735). For the peroxide fusion testing process, the samples underwent initial melting with sodium peroxide. Subsequently, depending on the type of the samples, it was digested in two acids and analyzed by the ICP-OES device. Finally, applying the mass coefficient allowed for the identification and reporting of certain oxide phases. To streamline the presentation of the extensive data, symbol identifiers were utilized in the tables (refer to Table 1).

Table 1. Symbol identifiers used in tables

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Symbol ID	Specifications
*	Elements with concentrations lower than the detection limit of the device in the prototype sample
@	Higher concentration, weight, and efficiency in comparative tables
#	Error of the device in measuring concentrations and its effect on efficiency calculations
$	Elements whose concentration has decreased to less than 0.01 ppm in one of the leaching stages
^	Concentrations higher than the detection limit of the device

3. Results and discussion

3.1. Qalkari

Qalkari is an industrial cupellation approach employed for initiating the recycling of e-wastes in Iran. In this section, the Qalkari furnace product and waste are scrutinized from morphological, chemical, and phase perspectives. This scientific information can serve as a valuable resource for scientists and technologists, enabling them to optimize the Qalkari variables, compare its efficiency with other pyrometallurgical processes used for the same purposes, assess the potential expansion of its applications, and design the subsequent hydrometallurgical processes. The SEM images of the Qalkari product and waste samples are presented in Fig. 4. It is evident that the product and residue contain chips ranging from 800 µm to 2 mm and irregular-shaped particles of 50-20 µm in size, respectively. The chipping process has effectively reduced the size of the Qalkari products and residues, enhancing the speed and efficiency of subsequent leaching due to the increased specific surface area [22-24].

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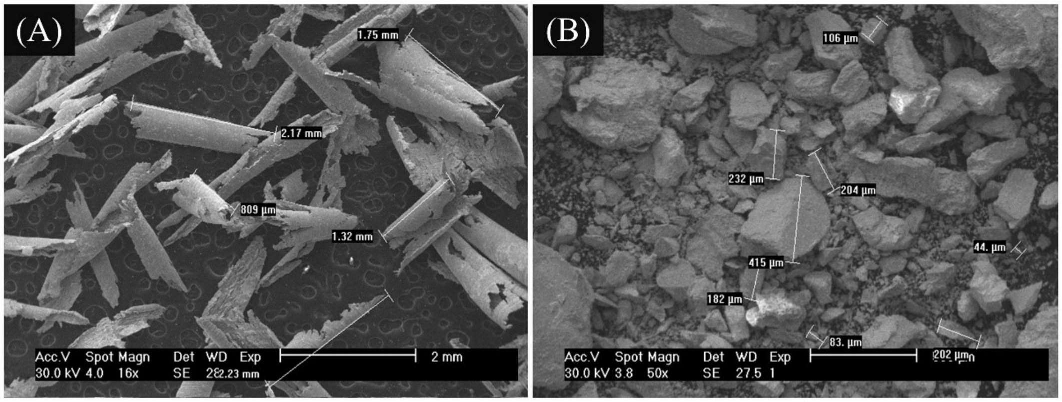

Fig. 4. SEM images of the Qalkari product shavings (A) and residuals (B).

The ICP-mass and XRF methods were used for the chemical analysis of the Qalkari product and waste samples. To minimize errors in the ICP-mass tests, an initial analysis of 68 elements was conducted, followed by proportional dilution for values outside the analysis range. For concentrations surpassing the accuracy range of this test (10,000 ppm), a supplementary XRF analysis was performed. Table 2 lists the results of the elemental analyses, with ppm indicating the amount of each element relative to others in the product or waste, and wt% denoting the partitioning of each element between the product and waste. It is worth noting that for 5 kg of the electronic wastes and 15.5 kg of the charged materials, the furnace output was 814 g Qalkari product and 17 kg waste, with the furnace dust excluded from analysis.

Table 2. Elemental composition in the product and waste of the Qalkari furnace

Element	Ppm		wt%		Element	ppm		wt%	
	Product	Waste	Product	Waste		Product	Waste	Product	Waste
Ag	12107.71	18.66	96.88	3.12	**Na**	299.22	2028.68	0.70	99.30
Al	701.36	1142.45	0.42	99.58	**Nb**	5.85	0.96	22.65	77.35
As	2.10	7.53	1.32	98.68	**Nd**	6.20	14.74	1.97	98.03

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Au	2407.72	*	100.00	*	**Ni**	1809.94	1580.31	5.20	94.80
B	51.66	108.03	2.24	97.76	**P**	1921.05	256.21	26.42	73.58
Ba	65.30	138.62	2.21	97.79	**Pb**	14412	840700	0.08	99.92
Be	*	0.10	*	100.00	**Pd**	155.95	3.03	71.11	28.89
Bi	604.68	14.68	66.35	33.65	**Pr**	0.48	2.81	0.81	99.19
Ca	582.85	1608.99	1.70	98.30	**Pt**	2.62	*	100.00	*
Cd	*	0.20	*	100.00	**Re**	*	0.12	*	100.00
Ce	82.20	47.80	7.61	92.39	**Rh**	9.85	0.64	42.25	57.75
Co	1.95	4.78	1.92	98.08	**Ru**	15.27	*	100.00	*
Cr	18.52	281.07	0.31	99.69	**Rb**	0.11	0.12	4.26	95.74
Cs	*	*	*	*	**S**	1601.36	1345.12	5.39	94.61
Cu	^	37882	^	^	**Sb**	8.10	219.89	0.18	99.82
Dy	0.70	1.30	2.52	97.48	**Sc**	1.63	2.21	3.41	96.59
Er	*	*	*	*	**Se**	0.88	*	100.00	*
Eu	*	*	*	*	**Sm**	0.19	0.14	6.08	93.92
Fe	282.65	2462.72	0.55	99.45	**Sn**	27.29	663.48	0.20	99.80
Ga	1.95	1.91	4.65	95.35	**Sr**	3.90	10.52	1.74	98.26
Gd	0.20	4.40	0.21	99.79	**Ta**	0.18	5.35	0.16	99.84
Ge	0.83	2.20	1.78	98.22	**Tb**	*	0.22	*	100.00
Hf	1.71	0.71	10.36	89.64	**Te**	31.19	7.65	16.34	83.66
Hg	*	0.18	*	100.00	**Th**	0.41	1.29	1.51	98.49
Ho	0.68	0.17	15.93	84.07	**Ti**	113.06	126.20	4.11	95.89
In	*	*	*	*	**Tl**	*	0.45	*	100.00
Ir	0.39	*	100.00	*	**Tm**	*	*	*	*
K	*	22.94	*	100.00	**U**	*	0.36	*	100.00
La	2.35	1.91	5.55	94.45	**V**	1.95	1.91	4.65	95.35
Li	*	*	*	*	**W**	14.49	7.59	8.37	91.63

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Lu	*	*	*	*	**Y**	1.27	0.47	11.40	88.60
Mg	65.30	131.93	2.32	97.68	**Yb**	*	0.11	*	100.00
Mn	2.92	33.46	0.42	99.58	**Zn**	29.24	122.37	1.13	98.87
Mo	0.97	2.87	1.60	98.40	**Zr**	10.72	11.47	4.28	95.72

Certain elements such as gold, bismuth, iridium, palladium, platinum, and ruthenium exhibited higher percentages in the Qalkari furnace product compared to the Qalkari furnace waste. The elevated concentration of copper in the product confirms that these elements are collected by copper. This process, concerning the initial product, is similar to the process reported in Refs. [25-27], resulting in the production of copper concentrates containing precious elements with purity close to blistered copper. In the Qalkari furnace waste, some elements like aluminum, arsenic, boron, and barium showed higher percentages than those in the Qalkari furnace product. These findings imply that many basic elements, or disturbing elements, are transferred to the waste during the Qalkari process. Notably, elements like gold, platinum, and ruthenium exhibited concentrations of less than 0.1 g in the Qalkari furnace waste. Furthermore, the amounts of silver and platinum in the Qalkari tablet were 31 and 2.5 times higher than those in the waste, underscoring the high efficiency of the Qalkari industrial process in differentiating between the product and waste.

The Qalkari operations closely resemble cupellation-based methods such as fire assay. Consequently, there are significant similarities between them in terms of governing mechanisms and the preferred distribution of elements in phases. Studies on the collection of Pd, Pt, Ir, and Rh using Cu [28-31] suggest the effective role of Cu as an Au and Ag collector in Qalkari. Due to its proximity to most precious metals in the periodic table, copper serves as a suitable solvent for precious metals, following the Hume-Rothery rules that consider factors like atomic size, crystal structure, electronegativity, and valence to form a solid solution. Additionally, variables such as time, temperature, ratio of flux compounds, and oxidizing environment influence the collection efficiency of elements [28, 29]. As the concentration of copper was beyond the measurement range, calculating the weight of each element based on the concentrations listed in Table 2 provides comprehensive information about the overall status of the outputs. In the Qalkari furnace product, the amount of copper and other non-measurable elements accounted for 96%, while that of the measurable elements was 4%. In the waste, the lead level was 84%, non-measurable elements constituted 11%, and the measurable elements made up 5%. Unmeasurable elements refer to those that could not be identified and measured by the ICP-mass and XRF tests.

The XRD profile of the Qalkari furnace product and waste specimens is depicted in Fig. 5. Three peaks of copper were identified in the product, and more peaks were recognized in the waste. It should be noted that there are overlaps in many peaks of the two phases of Pb (Cu_2O_2) and PbO. Due to the detection limitations of oxide phases in low concentrations by the XRD analysis, the Peroxide Fusion tests were also conducted (Table 3). This method allows the identification of ten oxide phases; however, it is not possible to identify some oxides, such as aluminum and sodium oxides. These results indicate that the oxidation process has generated the most significant phases of the Qalkari method.

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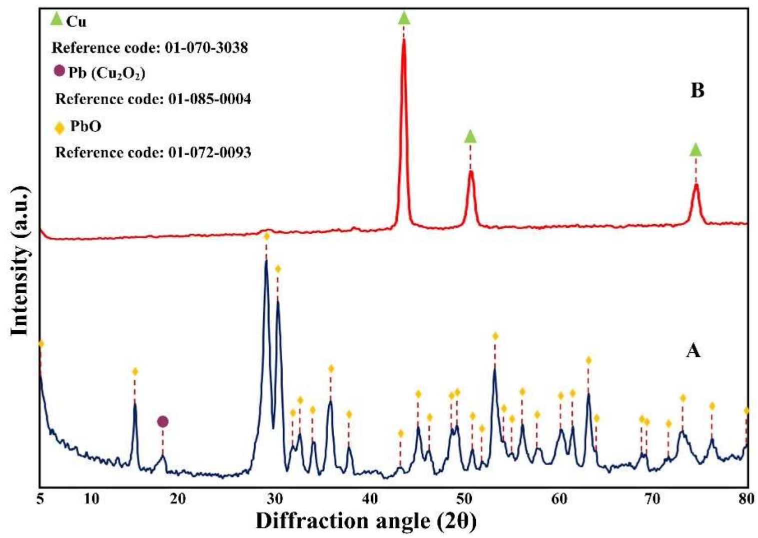

Fig. 5. XRD profile of the Qalkari waste (A) and product (B).

Table 3. Results of the Peroxide Fusion tests from the waste of the Qalkari furnace (in percent)

TiO_2	SO_3	P_2O_5	MnO	MgO	K_2O	Fe_2O_3	CaO	BaO	SiO_2
<0.05*	0.30	<0.05*	<0.05*	0.17	0.33	1.22	0.85	<0.05*	1.13

In the Qalkari process, metals undergo melting under oxidizing conditions provided by proximity to air, blowing oxygen, and high temperature. Molten lead is exposed to oxygen, transforming into litharge. Subsequently, litharge oxidizes other basic metals present, forming molten compounds through reactions with these oxides. Noble elements that do not react with litharge or oxygen, are then collected by copper and separated as concentrates of precious elements [32]. The oxidation process in Qalkari is enhanced by blowing oxygen onto the fuel and spraying it on the melting bed. Additionally, the discharge of the melt in several stages and its exposure to air during discharge contribute to the increased oxidation of the melt. While there are other industrial pyrometallurgical methods with an efficient recovery [27, 33, 34], the comparative advantages of Qalkari are its less complexity and potential to provide reusing its waste as an oxidizing agent in its next cycles. The latter feature is significant for saving litharge consumption and more effective waste management.

3.2. Gold Recovery

Qalkari effectively eliminates plastics and disturbing elements from electronic wastes, thereby enhances the efficiency of the subsequent hydrometallurgical stages used for the recovery of precious elements. This supports our hypothesis that the effective room-temperature recovery of gold, comparable to higher-temperature processes, is achievable through optimizing the other variables of hydrometallurgy. Accordingly, the G-1 phase of this section examines the influence of the leaching solution concentration on gold recovery at room temperature. Subsequently, in the G-2 phase, to further optimize the selected G-1 phase conditions, the impact of increasing the input solid amount on the dissolution rate at the same temperature is investigated. The significance of conducting the hydrometallurgical process at ambient temperature is underscored by the fact that higher temperatures lead to increased industrial costs. This is attributed to the requirement for more complex designs and more frequent inspections, repairs, and shutdowns of the plant.

Initially, all the samples in this section underwent an ICP-mass test for 68 elements, followed by a repetition of the analysis with proportional dilution for concentrations beyond the accuracy range. Upon completing the tests and filtering the solution, the sediment amount of each sample was measured to determine the dissolution efficiency, resulting in values of 0.027, 0.024, and 4.349 g for Specimens A, B, and C, respectively. Utilizing Eq. 1, the dissolution efficiency of the elements was calculated to assess the success rate of the samples in the G-1 phase, as tabulated in Table 4.

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$$\text{Dissolution efficiency of each element} = \frac{\text{Level of the element in the solution}}{\text{Level of the element in the specimen}} \times 100 \quad (1)$$

Table 4. Results of the dissolution efficiency calculations in the G-1 phase

Element	Solution A	Solution B	Solution C	Element	Solution A	Solution B	Solution C
Ag	99.99	100.00@	0.58	**Na**	98.22	132.04#	94.94
Al	62.10@	57.22	28.31	**Nb**	99.14@	99.14@	$
As	80.95	57.14	90.47@	**Nd**	100.00@	70.96	$
Au	$	$	$	**Ni**	99.84	123.08#	54.63
B	97.75@	93.10	83.81	**P**	100.00	240.55#	132.43#
Ba	99.08@	49.46	24.65	**Pb**	99/88	112.61#	86.83
Be	*	*	*	**Pd**	97.08	99.98@	2.69
Bi	99.93@	76.37	19.58	**Pr**	83.33@	83.33@	$
Ca	92.92@	59.48	36.64	**Pt**	95.41@	68.70	$
Cd	*	*	*	**Re**	*	*	*
Ce	99.87@	99.87@	$	**Rh**	99.49@	86.29	33.50
Co	97.43@	97.43@	$	**Ru**	100.00	84.47	30.12
Cr	98.81@	65.87	65.87	**Rb**	90.90@	90.90@	$
Cs	*	*	*	**S**	99.27	5.95	$
Cu	^	^	^	**Sb**	97.53@	88.88	54.32
Dy	100.00@	100.00@	$	**Sc**	98.15@	98.15@	$
Er	*	*	*	**Se**	90.90@	90.90@	$
Eu	*	*	*	**Sm**	99.67	100@	$
Fe	95.24@	95.20	63.96	**Sn**	79.48@	23.07	$
Ga	97.43@	51.28	$	**Sr**	#	#	#
Gd	100.00@	100.00@	$	**Ta**	$	$	$
Ge	96.38@	96.38@	$	**Tb**	*	*	*

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Hf	99.41@	93.56	$	**Te**	41.46@	24.39	$
Hg	*	*	*	**Th**	100.00@	100.00@	$
Ho	102.94#	88.23	$	**Ti**	$	$	$
In	*	*	*	**Tl**	*	*	*
Ir	76.92@	76.92@	$	**Tm**	*	*	*
K	*	*	*	**U**	*	*	*
La	97.87@	97.87@	$	**V**	100.00	100.00	$
Li	*	*	*	**W**	94.48@	86.61	$
Lu	*	*	*	**Y**	$	$	$
Mg	97.24	162.02#	97.24	**Yb**	*	*	*
Mn	92.46	92.46	95.89@	**Zn**	70.89	100.00@	91.41
Mo	103.09#	103.09#	$	**Zr**	99.99@	99.99@	$

As inferred from Table 4, some elements like magnesium, manganese, sodium, and lead exhibit a high dissolution level in all of the three solutions. This is significant when the removal or recovery of these elements is required without the need for heating and high acid usage. The investigation into arsenic reveals that increasing the acid concentration initially decreases and then increases its dissolution level. However, the dissolution level of zinc increases up to a level by increasing the acid concentration and then decreases. These behaviors can be attributed to various factors, including reactivity, deactivation (passivation), competitive dissolution of metals, and formation of complexes and ligands [35]. Experimental conditions such as temperature, concentration, and pH play a crucial role in these contributions, influencing the experiment's outcome [36-40].

While the gold recovery efficiency of 99.99% was obtained from all the three solutions, their leaching efficiency for the input solid differs, quantified as 99.73, 99.76, and 51.56% from

Solutions A, B, and C, respectively. The remarkable efficiency of Solutions A and B highlights the effectiveness of the prior Qalkari process in removing disturbing elements. Despite the relatively similar efficiency of Solutions A and B, the latter solution is concluded as the most successful solution of the G-1 phase due to its lower nitric acid consumption. It is noticeable that a small amount of impurity is introduced into the samples even from the elements with incomplete dissolution. For instance, the dissolution efficiency of chromium in Solution B is 65.87%. Given the ratio of chromium in the Qalkari furnace waste to the Qalkari furnace product is 316.98, the amount of this element is as small as 1.51×10^{-2} g in 814 g of the Qalkari tablets.

In the G-2 phase, to optimize the input solid amount at the selected solution of the G-1 phase (B), three samples were considered. The dissolution efficiency under Conditions G-2-1, G-2-2, and G-2-3 with 15, 20, and 40 g of the input solid, respectively, was 99.07, 75.39, and 59.21%, respectively. In the range of 10-15 g of the input materials, the difference in efficiency was insignificant (a tenth of a decimal), affecting only the grade of gold due to the non-dissolvable nature of this element in nitric acid. In conclusion, the removal of the disturbing elements during Qalkari enhances the efficiency of subsequent gold recovery, even in an on-step room-temperature hydrometallurgical process using nitric acid. Typically, 99.99% gold with a considerable purity measured by the ICP-mass analysis of the deposit was recovered without heating and using exclusive precipitants for gold. Additionally, the acid consumption and the input material amount are optimizable to enhance the purity and efficiency of recovered gold.

Comparison with similar studies on recycling of electronic wastes [16-18] and non-electronic wastes [41, 42] that did not employ any pyrometallurgical process before hydrometallurgy indicates that the dissolution level of some elements like Ag [16], Ag, Fe [42], Sn, Ag [18], Fe, and Zn [41] was less, whereas that of some like Al [18] was more than that obtained in this study. Furthermore, since gold is not dissolved in nitric acid, some studies aimed to purify gold and selectively recover it using other solutions and methods [18, 42]. However, the advantages of this study over other similar works lie in the selective recovery of gold in one step, the elimination of heating during the hydrometallurgical step, and the positive impact of the Qalkari process on the leaching time. The leaching time in this study was 30-60 min depending on the input materials, whereas it ranged from 2-5 h in similar studies where the ratio of the liquid to the input solid was greater than or equal to this study. Despite the use of microwave to reduce the leaching time in some recent methods like that reported in Ref. [43], its leaching time is not significantly different, considering that its input materials were refractory gold concentrates with a lower variety and amount of disturbing elements compared to electronic wastes. In conclusion, the removal of some disturbing elements during Qalkari, the reduction of the concentrate size during the chipping process (Fig. 4), and the increase in the acid concentration contributed to shortening the recovery time, highlighting the merit of the process used in this research.

3.3. Silver recovery

The objective of this phase is to compare three chlorine-based precipitants (HCl, NH_4Cl, and NaCl) for the room-temperature selective recovery of silver from the Qalkari-processed gold-recovered solution. The role of the prior Qalkari and room-temperature gold recovery in comparison to other pyrometallurgical and elevated-temperature gold recovery methods, respectively, in silver recovery is also analyzed. For this purpose, the solution samples

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underwent the ICP-mass analysis, with values outside the valid range being reanalyzed after proportional dilution, and concentrations with errors were removed. The comparison of the element concentrations before (Solution A) and after adding the precipitants (Table 5) reveals that some elements were precipitated along with silver. The silver recovery is 99.99% when using HCl, indicating the success of stoichiometric calculations in recovering the entire soluble silver level. However, compared to the other samples, this sample exhibits the highest sediment levels for boron, calcium, gallium, magnesium, manganese, neodymium, phosphorus, platinum, rhodium, ruthenium, antimony, and zinc. This issue suggests the presence of impurities, a low silver grade, and the removal of some precious elements like platinum along with silver from the solution. When using NH_4Cl, 99.99% silver is recovered, accompanied by the highest sediment amounts for bismuth, cobalt, chromium, iron, nickel, lead, and tin compared to the other precipitants. Additionally, it exhibits the lowest palladium deposition. Although these seven elements are considered impurities in silver, they can be readily removed by subsequent processes like electrolysis [44]. Notably, when palladium recovery is the focus, typically achieved using precipitants such as dimethylglyoxime or sodium formate [45, 46], the use of NH_4Cl is expected to enhance the recovery rate and purity of palladium by reducing the concentration of the disturbing elements in the solution. Using NaCl, 99.99% of silver is recovered, demonstrating the success of all the precipitants in the complete recovery of silver. However, this precipitant yields the highest sediment amounts for aluminum, cobalt, chromium, palladium, and tin. Examining these sediments reveals that this precipitator deposits fewer elements than the previous samples. However, in terms of the sediment type, palladium exhibits the highest amount compared to the other samples, suggesting this as a drawback of this precipitator for samples containing palladium.

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Table 5. Results of the precipitation efficiency calculations in the S phase

Element	HCl	NH_4Cl	NaCl	Element	HCl	NH_4Cl	NaCl
Ag	99.99	99.99	99.99	**Na**	0.00	0.00	0.00
Al	4.76	7.61	16.98@	**Nd**	40.32@	0.00	0.00
As	0.00	0.00	0.00	**Ni**	24.48	26.67@	25.39
Au	$	$	$	**P**	81.88@	0.00	0.00
B	72.07@	0.59	0.00	**Pb**	38.76	80.99@	77.66
Ba	0.00	0.00	50.07	**Pd**	10.17	6.47	35.53@
Bi	2.28	28.64@	20.93	**Pt**	44.00@	36.00	20.00
Ca	10.45@	8.30	0.00	**Rh**	45.91@	41.83	41.83
Ce	0.00	0.00	0.00	**Ru**	45.75@	35.94	33.33
Co	84.21	89.47@	89.47@	**Sb**	43.03@	36.70	27.84
Cr	26.77	33.33@	33.33@	**Sn**	41.93	77.41@	77.41@
Fe	16.12	29.71@	25.00	**Ta**	$	$	$
Ga	31.57@	0.00	0.00	**Te**	99.99	99.99	99.99
Ir	66.66	66.66	66.66	**Ti**	$	$	$
Mg	17.48@	0.00	0.00	**Zn**	57.89@	14.47	14.47
Mn	51.85@	0.00	0.00				

Given that the sedimentation rate of some elements in the ICP-mass test is not error-free despite repeated tests, it is not possible to select a successful precipitant based solely on the number of sedimented elements. Instead, the type of sedimented elements is a more critical factor for this purpose. Considering the relatively similar prices of these three types, it is inferred that each of these precipitators can be useful in the industry, depending on the type of existing elements and recovery goals. Nevertheless, ammonium chloride can be regarded as the overall best option, as it more effectively enhances palladium recovery.

In some other similar studies, silver has been recovered from solutions containing nitric acid and chlorine-based precipitants. Typically, the efficiency of silver recovery has been reported in the range of 4.84-100% within 30-180 min at temperatures of 25-90 °C in some studies on electronic waste recycling [19-21, 47] and non-electronic waste recycling [48, 49]. In contrast, the process utilized in this study offers a high efficiency without heating in only 30 min due to the elimination of some disturbing elements in Qalkari and the complete dissolution of silver in the gold recovery stage conducted prior to silver recovery. Additionally, despite the recovery of 99.93% of silver in 5 min by HCl from end-of-life photovoltaic panels in Ref. [50], there was an issue in terms of the precipitation of lead along with silver. This again emphasizes the significance of removing elements like lead before silver recovery, as conducted in this study by Qalkari.

4. Conclusions

This research was focused on the selective recovery of gold and silver from electronic wastes via a sequential process involving Qalkari and room-temperature hydrometallurgy. The specific outcomes of this study are outlined below:

1) Qalkari evidenced to be a valuable complement to hydrometallurgy, with its waste containing minimal amounts of gold, silver, and palladium as precious elements, alongside elevated levels of disturbing elements.
2) For gold recovery, optimizing the leaching solution concentration and the input Qalkari furnace product amount at 50% (v/v) and 15% (w/v), respectively, proved

effective in facilitating room-temperature hydrometallurgy with minimal acid consumption, quantified as a gold recovery of 99.99%.

3) HCl, NaCl, and NH_4Cl exhibited a similar efficiency for silver recovery from the nitric acid solution. However, for possible subsequent palladium recovery, NH_4Cl emerged as the most preferred precipitator for silver due to the lowest palladium deposition level along with silver.
4) The industrial significance of this study is to conclude the effectiveness of Qalkari in recycling various types of E-wastes simultaneously, the promising potential for the subsequent room-temperature hydrometallurgical recovery of gold and silver, and the priority of NH_4Cl in silver recovery for possible subsequent palladium recovery. The optimization criteria for these conclusions were to maximize product purity, process speed, and input materials level while minimizing the loss of the precious elements in the wastes and the acid consumption.

The following research directions can be inferred for the future work:

i) *Optimizing variables such as the type and level of loaded materials, concentration, temperature, pH, time, and precipitant type*: it can beneficially impact the recovery efficiency of precious elements because the reactivity of elements and their competitive dissolution and sedimentation are influenced by these thermodynamic and kinetic factors.

ii) *The scientific analysis of using Ag as a collector of precious elements in Qalkari despite its higher cost*: it is worthwhile to be investigated as it has potential to alter the characteristics of the Qalkari furnace product and waste. This idea is derived from the industry practice of using Ag as a collector for high-palladium samples, considering Cu as a disturbing element for palladium recovery in hydrometallurgical steps [51].

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