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Efficient Recovery of Silver and Aluminum from End-of-Life Photovoltaics: A Two-Step Leaching Approach

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Abstract: Recycling solar panels is crucial to mitigating the environmental impact of the growing volume of end-of-life photovoltaic waste and to conserve valuable resources, while achieving high purity in recovered materials ensures their effective reuse in the manufacturing of new solar panels, contributing to a sustainable and circular economy. This study proposes a two-step leaching process to recover Ag and Al from the silicon fraction of EoL photovoltaics (PVs). In the first-stage laboratory scale tests, 99% Al was extracted using 5% HCl at room temperature (19 °C) for 3 h The Al was precipitated as oxide from the solution. The silicon residue was then leached with 0.5 M HNO_3 at 85 °C for 2 h, extracting 99% Ag and producing Si with 99.83% purity. The silver was precipitated to produce metallic Ag with >99.9% purity. Then, 95.63% of Pb in the nitric acid effluent was removed using ion exchange resins. Further, methods to mitigate the effluent solutions were recommended and the entire process was presented in a flowsheet.

Keywords: recycling; solar panels; silver; aluminum; lead; leaching; recovery

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1. Introduction

The solar energy sector has experienced significant growth in recent years. Solar PV technology is now widely considered as one of the most promising routes to decarbonizing the energy industry and combating climate change. As the implementation of photovoltaic technology expands, so does the consumption of silicon, silver and other resources required in the manufacture of the solar panels. The rapid increase in solar panel deployment has led to a significant accumulation of end-of-life photovoltaic waste, posing challenges in waste management and resource recovery due to the complexity and hazardous nature of the materials involved, e.g., lead [1]. The current production capacities of the resources will not satisfy current market demand, especially with regard to fulfilling the Paris agreement climate goals. Further, although solar panels have a user-life of 25–30 years, upgrades in technology have made way for a build-up in waste of discarded or damaged panels and this will only become worse in the foreseeable future [2]. This situation requires efficient resource management [3] and, more importantly, cradle-to-cradle recycling methods that will see the bulk of the materials recycled back into the manufacturing of new panels. Currently, there is no recycling process that fulfills these cradle-to-cradle principles. The RETRIEVE (www.retrieveproject.eu) project aims to solve this challenge by innovating the circularity in the PV industry via developing and improving state-of-the-art recycling processes in the upstream industry. RETRIEVE will develop cradle-to-cradle recycling processes for components of end-of-life (EoL) PV panels that will upcycle their quality to meet the current requirements of newly manufactured Made in Europe PV panels.

As part of this project, this study focuses on the recovery of silver (Ag) and aluminum (Al) from the cell fraction along with recommended methods for recovering valuable metals from EoL silicon. Thus far, several studies [4–12] have been carried out on metal recovery

from recycled PV panels at lab and at pilot scale. The focus was generally on recovering silicon, copper, and aluminum, as silver recovery needs intensive extractive metallurgical methods [13]. Silver has the most economic value among the metals. However, the handling of other metals, particularly Pb, is of great importance as it is toxic and hence an environmental threat. To establish an innovative, environmentally friendly, and high-yield process for metals, the regeneration and re-use of process chemicals must also be considered along with the mitigation of hazardous impurities. The study will evaluate the leaching and recovery of Ag and Al, combined with methods to remove Pb, resulting in a flowsheet that not only recovers the valuable metals and removes hazardous elements, but one that is innovative, environmentally friendly, and sustainable; incorporating process chemicals can be possibly regenerated for re-use or processes that produce useful by-products.

Nitric acid is widely used in the leaching of silver from various feed materials [14–17]. It is proposed that the Ag is leached in nitric acid, recovered as AgCl, and reduced to metallic silver using hydrazine in the presence of NaOH [16–18]. The process occurs according to Equations (1)–(3).

$$Ag_{(s)} + 2HNO_{3(aq)} \rightarrow AgNO_{3(aq)} + NO_{2(g)} + H_2O_{(aq)} \quad (1)$$

$$AgNO_{3(aq)} + HCl_{(aq)} \rightarrow AgCl_{(s)} + HNO_{3(a)} \quad (2)$$

$$2AgCl_{(s)} + N_2H_{2(l)} + 2NaOH_{(aq)} \rightarrow 2Ag_{(s)} + 2NaCl_{(aq)} + N_{2(g)} + 2H_2O_{(aq)} \quad (3)$$

The above process results in the co-leaching of other elements like Al, and specifically toxic ones like Pb. As a result, sequential separating and recovery techniques are employed to recover the principal elements. This usually means using NaOH to precipitate Al and H_2SO_4 to recover Pb. The addition of these chemicals means it is not possible to reuse the HNO_3. This study will therefore explore a two-stage leaching system in which a pre-leach step will be used to extract Al prior to the leaching of Ag. This will allow for the separate recovery of Al and the reuse of the HNO_3 when the Ag is recovered. Various organic acids, ammonia, Na_2CO_3, H_2SO_4, and HCl were evaluated using OLI process simulation software (https://www.olisystems.com) and small-scale test work. This early campaign revealed that HCl was the most promising reagent to investigate further for Al leaching. Furthermore, the study also investigates the use of ion exchange (IX) resins in removing Pb from solution in favor of the conventional method of $PbSO_4$ precipitation [19,20]. The IX resins do not add chemicals to the process, and removing the Pb from the resins with acid regenerates them for continued use.

2. Materials and Methods

The samples used for test work were silicon solar cells (Figure 1a) manufactured and supplied by the Soltek department at the Institute of Energy Technology (IFE) in Kjeller, Norway. The solar cells were milled down to 400 µm (Figure 1b) for test work and analyses. The rectangular solar cells were milled to increase surface area and facilitate stirred leaching tests and digestion of the samples for analyses. The chemical composition of the solar cells was identified as follows: a sample of the milled cells was completely digested in acids and afterwards diluted for analysis by high-resolution inductively coupled plasma mass spectrometry or ICP-MS (Element XR, Thermo Fisher Scientific, Dreieich, Germany). The same procedure was used to quantify the elements in leached solar cell samples. Table 1 shows the main elements, except for silicon, oxygen, nitrogen, and possibly other very minor elements. The elements of significance were Al, Ag, and Pb.

Table 1. Analysis of elements in solar cells as weight percentages (wt%).

Si	B	Al	P	Fe	Ni	Zn	Zr	Ag	Sn	Pb
83.67	0.007	14.15	0.001	0.007	0.002	0.004	0.001	1.95	0.001	0.21

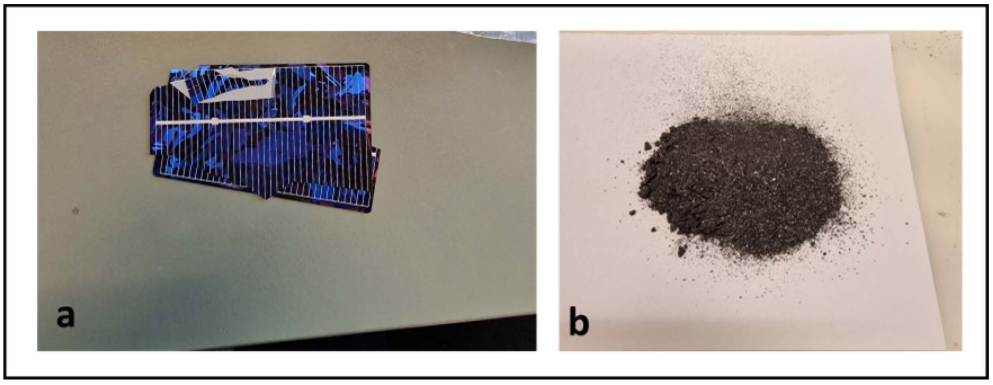


Figure 1. (**a**) Solar cells. (**b**) Ball-milled solar cells.

Test parameters were largely selected from the literature but were developed to add new values as the test campaign proceeded. All leaching tests (Table 2) were conducted in a three-neck 250 mL round-bottomed flask using 200 mL of solution. Technical reagent-grade 37% HCl and 65% HNO_3 were used to make all the leaching solutions. Necessary precautions were taken when working with both acids, such as wearing the required personal protective equipment (PPE) and preparing all acid solutions and conducting experiments in a fume hood. Then, 20 g samples of ball milled solar cells were leached with the desired solution, with the mixture agitated using a magnetic stirrer and heated to the desired temperature (Table 2) using a heating mantle. For tests at temperatures higher than room temperature (19 °C), the temperature was monitored using a thermometer while the slurry volume was maintained using a water-cooled condenser placed at the central neck of the flask. For the tests at temperatures higher than room temperature, the time it took for the temperature to reach the desired point was included in the duration of the test. Samples were withdrawn and syringe-filtered with a 0.45 µm membrane at various intervals of each test to be analyzed via X-ray fluorescence (XRF) to determine the extent of metal extraction at that point. After each analysis, the sample was returned to the flask. On completion of the tests, the slurry was allowed to cool when necessary, and filtered using a porosity 4 funnel filter, and then the filtrate was further filtered using 0.45 µm syringe filters. While still in the filter funnel, the solid residues were washed thoroughly by filtering through tap- and then finally distilled water. The residues were either immediately used in the next leaching stage (from HCl leaching) or oven-dried at 65 °C overnight at the end of the second leaching stage for the determination of the element content (Ag, Al, or Pb). Either ICPMS, XRF, or X-ray diffraction (XRD) was used for this quantitative elemental analysis.

Table 2. The various parameters explored for the first-stage HCl leach and the second-stage HNO_3 leach over several tests conducted.

Process	Concentration	Temperature	Time
HCl	5 and 10%	19 and 60 °C	2–4 h
HNO_3	0.5, 2 and 4 M	70 and 85 °C	2–4 h

Metal extractions were calculated using the following two methods:

1. By using the amounts extracted by solution, and calculating extraction as a percentage of the amount in the unleached sample:

$$[(C_E \times V_L)/M_U] \times 100$$

C_E = Concentration of element in solution [g/L];
M_U = Amount of metal in unleached sample [g];
V_L = Volume of leaching solution [L].

2. By analyzing the solid residues to determine the amounts left in the residue

$$[(M_U - M_L)/M_U] \times 100\ [\%]$$

M_L = Amount of metal in leached sample [g].

For the Al recovery tests, the leachate from the HCl leach was placed in a beaker with a pH probe. Granular NaOH was added 2 g at a time, while the pH was monitored. Solution samples were analyzed via XRF at various intervals to determine when all the Al had been precipitated. When this was achieved, the slurry was filtered using a porosity 4 funnel filter. While in the filter, the Al precipitate was washed with water, collected in a beaker, and oven-dried at 65 °C overnight for XRF and XRD analysis. The filtrate was further filtered using a 0.45 μm syringe filter for XRF analysis. This was to further confirm the Al had been completely precipitated.

Ag recovery from the HNO_3 leachate was conducted by adding 1 mL of 37% HCl at room temperature to the leachate in a 250 mL beaker. This created a milky solution which was allowed to settle for for 4 h for easy sampling. A sample was taken from the supernatant solution, filtered using a 0.45 μm syringe filter, and analyzed via XRF. The analysis confirmed that most of the Ag had precipitated. The supernatant was allowed to sit over night before being decanted to separate it from the AgCl precipitate, and further filtered with a 0.45 μm syringe filter. The precipitate was washed with water. A solution of 5 mL hydrazine and 2 g of NaOH dissolved in 50 mL of deionized water was prepared. The solution was added to the AgCl in a beaker and stirred for a few minutes. This was used to reduce the silver chloride to metallic Ag. The solution was decanted, and the solid residue washed thoroughly with distilled water. It was then oven-dried overnight at 65 °C for analysis via XRF and XRD.

Pb removal from the decanted and filtered HNO_3 solution above, after the Ag precipitation, was investigated using Puromet™MTS9300H resin, manufactured and supplied by Purolite, an Ecolab Company (Paris, France). The pH of the leachate was adjusted to 3-4 using granular NaOH. Briefly, 50 mL of leachate was placed in an Erlenmeyer flask. Then, 1 g of resin was added, and the flask was shaken at a rate of 600 rpm using a platform shaker for 1 h. A sample was withdrawn and analyzed via XRF to determine the extent of Pb removal.

3. Results and Discussion

Of several tests conducted, results presented and discussed will mostly be from two tests (Sample 1 and Sample 2). Samples 1 and 2 gave the best results regarding parameters of reagent concentration, time, and temperature, and were most consistent when experiments were repeated. The HCl pre-leach achieved nearly 100% extraction of Al in tests, as confirmed by XRF analysis of the solution. Using a 5% HCl (Sample 1) requires more time than using a 10% HCl (Sample 2) solution, but shows the same extraction results of Al (Figure 2).

Analysis of the leached residues after the HNO_3 leach showed that up to 99% of the Al had been leached in both cases. It should be noted that it was not possible to analyze the residue after the HCl leach, as at least half the sample was required for the analysis. Further, the material could not be returned for the second stage leach test. Negligible quantities of Ag in the range of 20 ppm in solution were leached, along with some Pb in the range of 70 ppm in solution. It was noted that at higher temperatures (50 °C), more Pb was leached; however, in the follow-up HNO_3 acid leach, Ag extraction was considerably poorer. It is hypothesized that at high temperature, a small amount of the Ag is leached and immediately precipitates as insoluble AgCl, coating the rest of the Ag. This passivates the Ag and prevents it from leaching in the HNO_3 acid. It was thus decided that the optimal leaching temperature for the first stage was room temperature (19 °C). For the Al precipitation using NaOH, it was found that all the Al precipitated at a near-neutral pH of

6.54. For the nearly 200 mL of HCl leachate solution, 12 g of NaOH was required to achieve this pH and precipitate all the Al. The process was suspected to occur as follows:

$$AlCl_{3(aq)} + 3NaOH_{(aq)} \rightarrow Al(OH)_{3(s)} + 3NaCl_{(aq)}$$

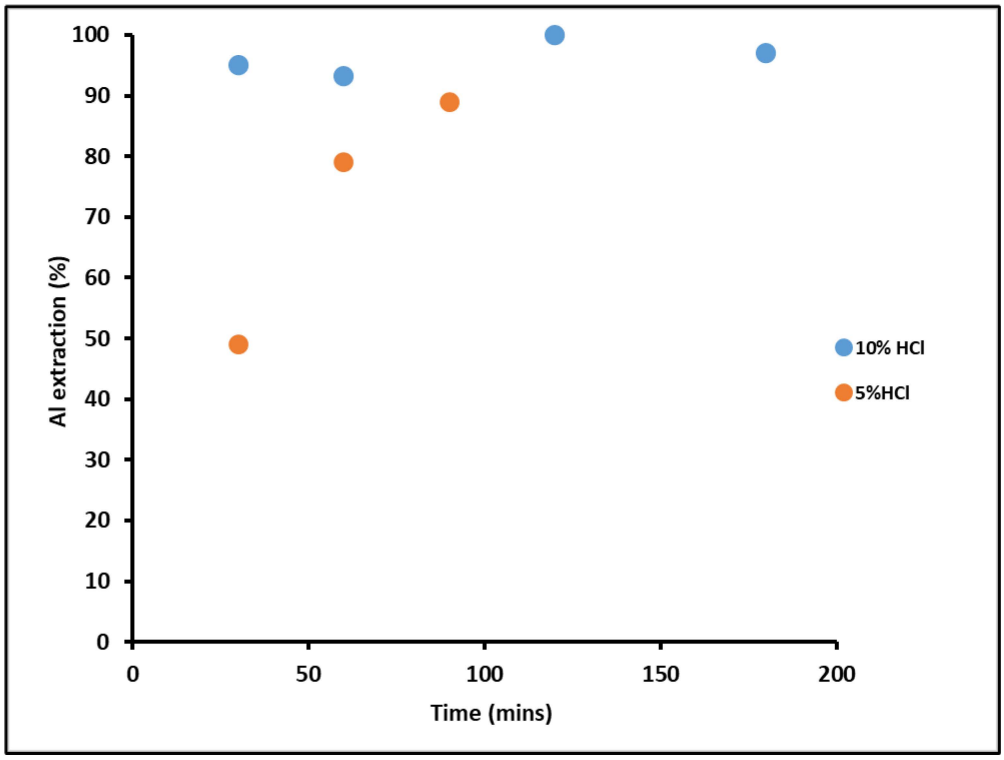


Figure 2. Al extraction in 5% (Sample 1) and 10% (Sample 2) HCl leaching tests from milled solar cells at room temperature (19 °C).

XRF analysis confirmed the presence of Al with some Cl and possible Fe, Cu, Zn, and Pb impurities, which could not be quantified. From this, it was suspected that the precipitate was $AlCl_3$ with some impurities. However, XRD analysis showed that the precipitate was composed of mostly Al-oxides, with possible contamination of Cl, Fe, Cu, Zn, and Pb (Figure 3a). It should be noted that except for Pb, the other elements were present in the HCl leachate solution in quantities below the detection limit, according to the XRF analysis. Further, the XRF analysis of the solution after Al precipitation did not detect any heavy elements. It is thus concluded that the solution is mostly a brine. It might be possible to consider exploring reverse osmosis (RO) to recover the water and produce NaCl as a saleable product. This process can be referred to as desalination and has long been established at industrial level [21,22]. The XRF analysis also showed no heavy metals in the solution. This means that the water produced from the proposed RO process can be recycled back into the main process, specifically in HCl leaching. At a larger operating scale, if heavy metals are present and they accumulate to high levels or it is decided that the water needs to be discharged, methods such as precipitation, ion exchange resins, solvent extraction, or adsorption [23] can be explored to remove them.

In the HNO_3 leach, XRF analysis of the solution samples confirmed that 0.5 M HNO_3 at 85 °C for 2 h was sufficient to extract 99% of the Ag. This was further confirmed by analysis of the solid residue samples (Table 3).

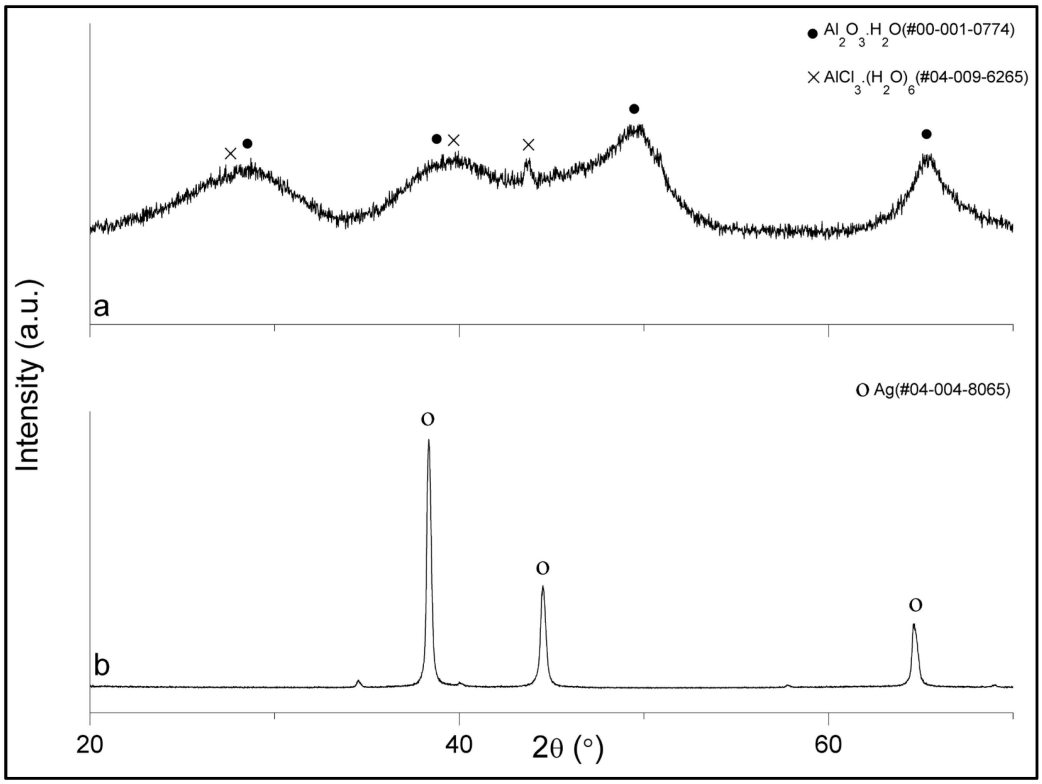


Figure 3. XRD micrograph of (**a**) the Al-compounds precipitated using NaOH and (**b**) the metallic Ag produced by using hydrazine as a reducing agent.

Table 3. Analysis of the non-treated sample and solid residues of two different samples using digestion and ICP-MS, showing metal concentration, the corresponding weight percentage, and the extraction percentage achieved.

	Ag (ppm) in Sample	Ag (%) in Sample	Ag Percentage Extraction Achieved	Al (ppm) in Sample	Al (%) in Sample	Al Percentage Extraction Achieved
Untreated Si cell	19,496	100.00%		141,467	100.00%	
Sample 1 (treated)	381	1.95%	98.05%	1201	0.85%	99.15%
Sample 2 (treated)	248	1.27%	98.73%	1780	1.26%	98.74%

The leaching patterns were identical for both Samples 1 and 2 as constructed from the XRF analysis of the solution samples. Figure 4 shows the leaching pattern for Sample 2, where it is seen that a dramatic increase occurs in Ag extraction from 55 to 72 °C. Some co-leaching of Pb in the concentration of 160 ppm was also experienced. However, Ag was the element leached in the most abundance in the range of 1300 ppm. Furthermore, there was around 280 ppm Al in the leachate solution. This varied with different tests, as some did not find any. This small amount of Al compared to the HCl leachate, which had 14,000 ppm, confirms that the bulk of the Al is leached in the HCl step even though the residue could not be analyzed after that leaching step, as a large portion of the residue is required for the analysis. The follow-up precipitation process with HCl use recovered 99% of the Ag in solution based on an XRF analysis of the solution before and after precipitation. Overall, this represents a 98% recovery of Ag from silicon. Afterwards, the reduction of precipitated AgCl to metallic Ag was carried out using hydrazine. Detailed information can be found in Section 2. The reduction process produced a product that, according to XRD analysis, was metallic and mostly silver (Figure 3b). XRF analysis showed the Ag was of >99.9% purity with some possible contamination of Pb and Fe. Around 160 ppm Pb precipitated, based on the XRF analysis of the solution after precipitation. The resulting

effluent is thus mostly regenerated and unreacted HNO_3, as per Equation (1), and 160 ppm of Pb. It is worth noting that the solution can be re-used in the process. When the Pb accumulates to unacceptable levels, or it is decided to discharge the solution, the IX resin system can be used to remove the Pb. This study achieved successful Pb removal using the Purometᵀᴹ MTS9300H resin. XRF analysis of the solution showed that the concentration dropped from 160 ppm to 7 ppm after an hour of treatment with the resin. This represents 95.63% of the Pb recovered.

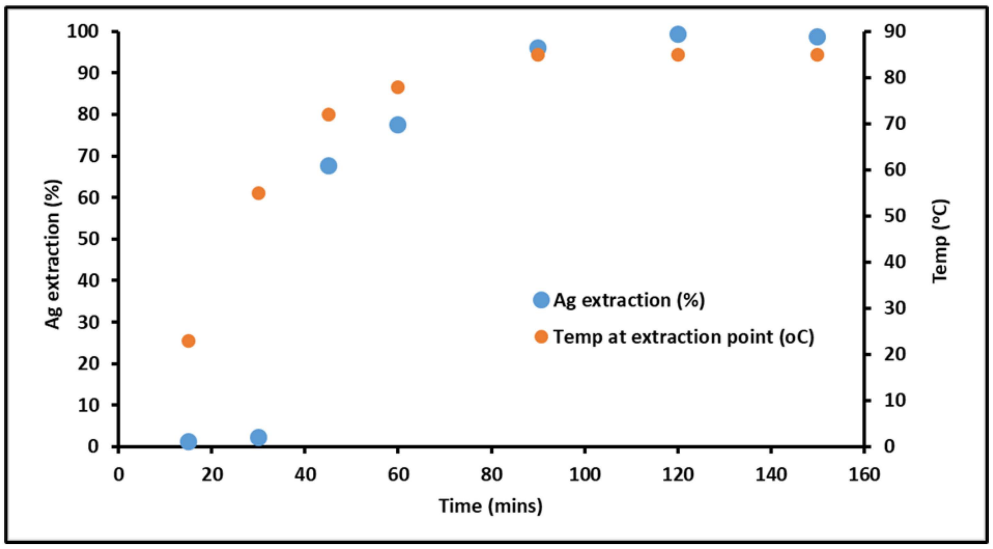


Figure 4. Leaching of Ag from HCl leached residue (Sample 2) using 0.5 M HNO_3 at 85 °C.

The above experimental results are presented and summarized in a flowsheet, Figure 5, and in Table 3. The flowsheet incorporates elements tested and proven in the laboratory, such as Ag and Al extraction and recovery and Pb removal. Other elements of the flowsheet are conceptual and are yet to be tested, such as the RO stage, heavy metal removal, and Pb stripping and resin regeneration.

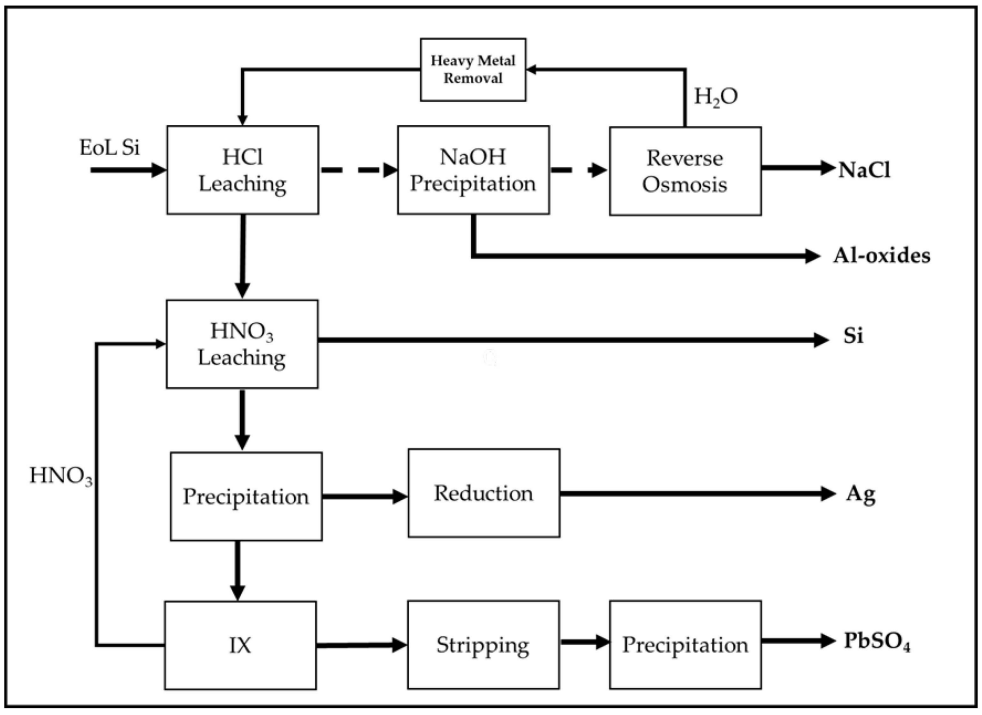


Figure 5. Proposed two-step process for extracting Ag and Al from Si of EoL PVs.

4. Conclusions

This study demonstrates a two-step leaching process for efficiently recovering silver (Ag) and aluminum (Al) from the silicon (Si) of end-of-life (EoL) photovoltaic (PV) panels, resulting in the development of a conceptual flowsheet. In the first step, Al is leached using 5% HCl at a low temperature for 3 h, achieving a 99% recovery rate. The Al is precipitated as Al-oxides using NaOH, generating a brine effluent solution that could potentially be treated with reverse osmosis to produce NaCl and water for recycling within the process. The remaining Si residue is then leached with 0.5 M HNO_3 at 85 °C for 2 h to extract 99% Ag, which is subsequently recovered by precipitation with HCl and reduced to metallic Ag with a purity of >99.9% using hydrazine, achieving an overall Ag recovery of 98%. Additionally, lead (Pb) can be effectively removed from the HNO_3 solution after Ag precipitation using Purometᵀᴹ MTS9300H resins.

The integration of sequential leaching with ion exchange resins for Pb removal provides a novel approach that enhances metal recovery while minimizing environmental impact, distinguishing this method from existing recycling techniques. Although chemical regeneration is proposed for future research to further reduce waste and operational costs, the current study lays a strong foundation for sustainable, large-scale PV recycling. The promising results highlight the need for further evaluation and optimization through additional laboratory work to confirm the scalability and economic feasibility, aligning with circular economy principles and sustainable resource management.

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Data Availability Statement: The original contributions presented in the study are included in the article, further inquiries can be directed to the corresponding author.

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