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# Selective leaching for the recovery of Valuable metals from waste crystalline-silicon solar cells

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## Abstract

Due to the rapid expansion of the solar energy market over the past few decades, a large amount of waste from discarded silicon-based solar modules has accumulated, representing a considerable source of recoverable silicon and silver. High-efficiency metal recovery from solar cells in the recycling process is achieved through leaching with sulfuric, hydrochloric, and nitric acids; numerous studies have shown that nitric acid is particularly effective in recovering metals including aluminum, silver, copper, and lead from these cells. The selective leaching of valuable metals from pulverized silicon-based solar cells was examined in this study, employing nitric acid to achieve the dual objective of recovering the leached metals and simultaneously purifying the undissolved silicon fraction to yield high-purity silicon. For the leaching experiments, nitric acid solutions of 1M, 2M, and 3M concentrations were prepared as molar solutions and then treated in an ultrasonic cleaner, which was kept at a constant temperature of 60 °C for the duration of the experiment. The effect of leaching time on the further purification of silicon through selective leaching using a 1M concentration at 60 °C was also investigated. Throughout the duration of the leaching experiments, analysis of the silicon phases within the remaining material following the selective leaching process, with careful consideration given to both the concentration of the leaching agents and the length of the reactions, verified that the metals were effectively removed only after the leaching process had surpassed a 30-minute period. With a purity exceeding 99%, the silicon is of exceptionally high quality, indicating that advanced processing techniques were employed. Silver extraction from the solution was accomplished using copper, a reducing agent, introduced into a silver nitrate solution; this initiated a redox reaction, with varying amounts of copper (0.25, 0.5, 0.75, and 1 gram) added according to stoichiometric calculations based on the reaction between the silver nitrate solution and the solid copper. Silver precipitation occurred at 0.23 and 0.55 grams, respectively, upon the addition of 0.75 and 1.0 grams of copper, which caused the precipitation reaction.

**Keywords:** Copper Addition, Copper Substitution Reaction, Pulverized Solar Cell, Silicon Purification.

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## 1. Introduction

In the last few years, the global solar power generation market has seen an explosion in growth, driven by several key factors: the increasing global demand for energy, substantial government support programs and policies, growing concern about environmental pollution, and a corresponding exponential increase in the installation of solar modules [1, 2] By 2019, the global cumulative installation of solar cells had surpassed 500 gigawatts, a figure projected to exponentially increase to over a terawatt by 2023, based on the findings of Fatih [3] and Shoaib, et al. [4]. Due to this market expansion, electricity can now be produced at a significantly lower cost compared to other energy sources, particularly in areas with advantageous climates, thus making it a more economical and environmentally friendly option. Given the typical twenty to thirty-year lifespan of solar modules, and the evidence from studies by Ga-Min and Hyo-Sik [5] and Kyounga and Cha [6] showing that many have reached the end of their operational lives, partial scrapping and demolition are currently being carried out. It is projected that by the year 2030, the amount of waste generated from solar modules will surpass a significant 1.7 million tons, according to various studies and reports by Tan, et al. [7]; Iliana, et al. [8] and Georgia, et al. [9]. In light of the anticipated growth in the number of waste solar modules in the coming years (as indicated in references as Kabir, et al. [10] and Dheeraj and Sushil [11] the European Union (EU) has called for the implementation of recycling programs designed to handle renewable energy wastes, and specifically, for the categorization of discarded solar modules as electrical and electronic waste. A comprehensive overview of the solar module market reveals that over ninety-five percent of modules installed worldwide are based on crystalline silicon, a technology encompassing a variety of components including glass, aluminum frames to house the solar cells, PV ribbons, EVA encapsulant for protection, and junction boxes for connection [12]. The high cost of purifying polysilicon (poly-Si) for solar cells—representing over 30% of solar module production costs—highlights the economic potential of recovering and reusing pure silicon reclaimed from end-of-life solar cells in order to enhance the sustainability and cost-effectiveness of solar module manufacturing processes. Furthermore, the importance of silver in solar cell technology is underscored by its significant role in facilitating the transfer of electrical charge—a key function that involves conducting electricity from inside the solar cell to the electrical system, as evidenced in the literature [13-15]. It has also been reported that a life cycle assessment (LCA) of the environmental impact of the raw material acquisition for solar cell manufacturing (specifically, 2N grade silicon obtained from quartz and sand) indicates that silicon, calculated per square meter, possesses the second-largest global warming potential among all evaluated factors, according to study by Lizabeth, et al. [16]. Several studies have investigated relatively low-temperature recycling techniques for solar cells, employing various acids to recover valuable metals like silver, aluminum, and silicon, thereby improving cost-effectiveness and enhancing solar module manufacturing processes. In this study, we propose a novel method for the recycling of silicon-based solar cells involving selective leaching using nitric acid to separate metals and silicon from pulverized solar cells, followed by the recovery of silver through a substitution reaction with copper in the resulting silver nitrate solution—a process designed for industrial applications in solar cell recycling.

The majority of recent research has investigated methods for recovering valuable metals from spent solar cells, with a focus on characterizing how components of these cells leach when treated with various acids including sulfuric acid, hydrochloric acid, and nitric acid. Because aluminum, the most abundant component in solar cells, is highly susceptible to leaching in sulfuric, hydrochloric, and nitric acids, its potential for dissolution in the form of silicon compounds when exposed to sulfuric acid is a significant factor to consider. It is expected that silver, copper, and lead will dissolve into hydrochloric acid to form a MeCl precipitate, however, studies by Tembo, et al. [17] and Qi, et al. [18] have shown that the copper and lead present in the PV ribbon do not oxidize in sulfuric acid. Nitric and hydrochloric acids are acceptable to recover metals such as Al, Ag, Cu and Pb from solar cells according to the reports of studies by Jeongeun, et al. [19]; Youn, et al. [20] and Dina, et al. [21]. Several studies Chen, et al. [22]; Eleni, et al. [23] and Dias, et al. [24] have investigated low-temperature acid leaching of silver from solar cells using nitric acid, and these studies indicate that the optimal $HNO_3$ concentration ranges from 2 to 10 molar solutions. De Oliveira, et al. [25]; Kuczyńska-Łazewska, et al. [26] and Huang, et al. [27] found in their studies that a 2 and 3 M nitric acid solution could effectively leach silver from solar cells at a low temperature, with subsequent silver recovery being achieved by employing a two-step process involving chemical precipitation and a final reduction step. As a result of the research goals, this study employed nitric acid as the leaching agent for extracting silver from the powdered silicon-based solar cells under investigation. As shown in Scheme 1, the experimental process conducted in the laboratory focused on optimizing three key parameters–solution concentration, leaching time, and copper substitution–to improve the efficiency of metal leaching and recovery. In an effort to determine the quality of silicon obtained from the processing of silicon–based solar cell powder, further studies were undertaken to investigate and analyze silicon purification techniques. To further investigate the precipitation of silver from solution into a solid phase during a substitution reaction with copper powder, additional experiments were conducted to determine the relationship between the amount of copper added and the extent of silver precipitation. According to our findings, the leaching method studied in this research can effectively recover metals, leading to the possibility of reusing discarded solar panels in industrial settings.

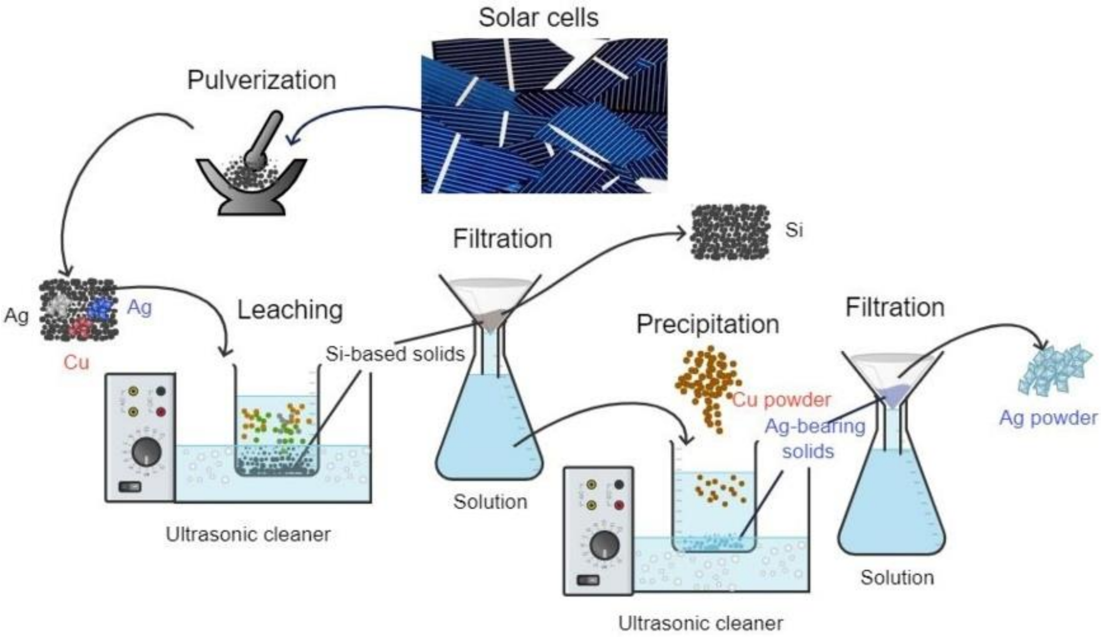

**Figure 1.**
The schematic diagram of the leaching processes conducted under the specified conditions, coupled with precipitation techniques, the recovery of silver can be influenced in a way that facilitates a cost-effective method for obtaining high-grade silicon and a significant amount of silver.

## 2. Materials and Methods

### *2.1. Experimental Materials*

The solar cell used in this investigation was composed of three materials: silicon, which is commonly used in solar cells, and the metals silver and aluminum. Solar PV modules are comprised of solar cells, which are sandwiched between layers of EVA film and encased within an outer structure that includes a glass front, back sheet, junction box, and an aluminum frame. Utilizing a high alumina mortar and pestle, the solar cells underwent a pulverization process, resulting in a fine powder. X-ray fluorescence (XRF) analysis revealed the solar cell powder's composition to be primarily silicon (89.74 wt.%), with a substantial aluminum content (10.17 wt.%), and a small amount of silver, as detailed in Table 1.

**Table 1.**
The composition of the pulverized solar cell by XRF.

| **Element, wt.%** | | |
|---|---|---|
| **Si** | **Al** | **Ag** |
| 89.7483 | 10.1776 | 0.0742 |

As depicted in Figure 2, the solar cell fragments were separated from the solar PV module through a process of careful mechanical and physical treatment. The solar cells had a PV ribbon attached to them; this ribbon was also removed from the solar cells. A SEM-EDS mapping analysis was conducted to investigate the elemental composition on both the front and back surfaces of the solar cell, as shown in Figure 2a and 2b. The presented EDS maps reveal the elemental distribution on both the front and back sides, indicating a front side layer primarily composed of silicon (approximately 40%), silver (approximately 48%), and a smaller amount of aluminum (0.43%), while the back side layer consists mainly of aluminum (approximately 36%), silver (approximately 28%), and a significantly smaller percentage of silicon (approximately 4%).

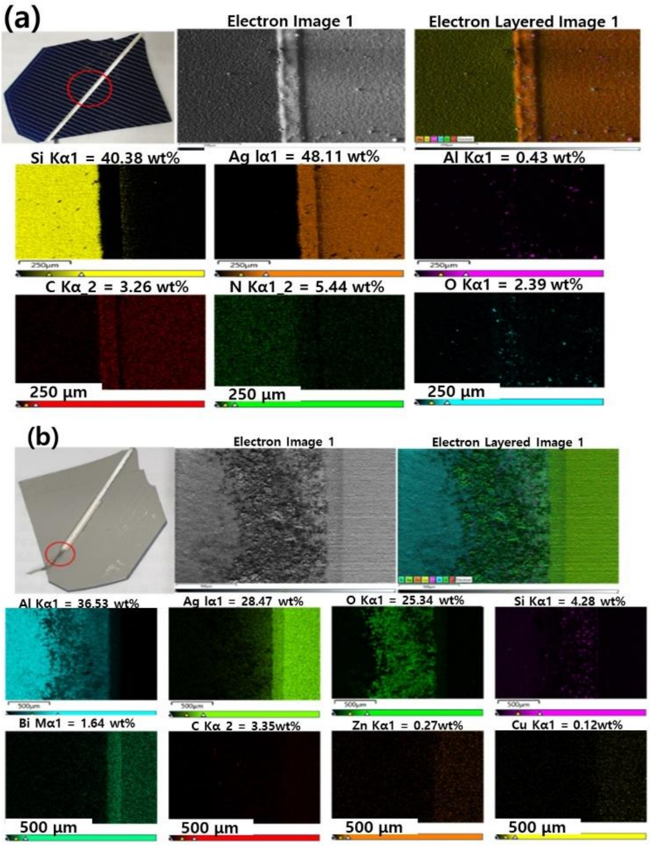

**Figure 2.**
SEM image of solar cell. (a) front side, (b) back side.

In order to identify the components of the solar cell powder, an XRD analysis was performed, which involved a thorough analysis. Based on the XRD analysis shown in Figure 3, the composition of the solar cell powder was determined to be a mixture of silicon and aluminum.

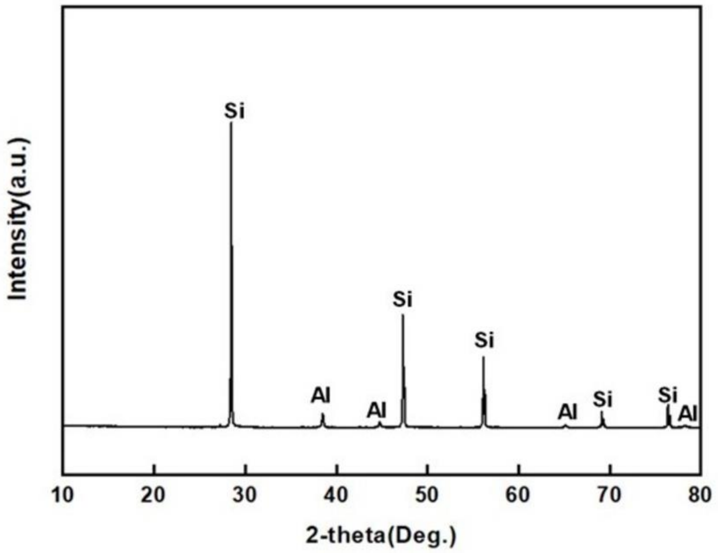

**Figure 3.**
XRD pattern of the pulverized solar cell.

### 2.2. Analysis Methods

The chemical composition of the pulverized solar cell was determined using a Shimadzu XRF-1800 model X-ray fluorescence spectrometer; this method provided a highly accurate and precise analysis of the concentrate's chemical makeup, yielding highly accurate results. The sample, having been placed with careful attention to detail into a platinum crucible, was then loaded into the XRF machine to begin the process of analysis. To precisely quantify the metallic components present, a comprehensive analysis was performed utilizing the Agilent 5800 ICP-OES, a highly advanced piece of equipment known as an Inductively Coupled Plasma Optical Emission Spectrometer, which is designed to measure the amounts of metals with a high degree of accuracy. The mineral composition of the sample and its precipitates was established through the use of a state-of-the-art X-ray Diffractometer, specifically the X'Pert-MPD PANalytical model, which employed a high-intensity 3 kW Cu-Kα X-ray tube to facilitate the analysis and yield conclusive results. During the XRD data acquisition for the sample, a comprehensive scan encompassing an angular range from 10 to 80 degrees was performed over a 10-minute period, utilizing incremental steps of 0.02 degrees for enhanced resolution and data accuracy. The Central Laboratory at Pukyong National University provided the facilities for the execution of all material analyses, which included X-ray fluorescence (XRF) spectroscopy, inductively coupled plasma optical emission spectrometry (ICP-OES), and X-ray diffraction (XRD). For a complete examination to determine the morphology and elemental distribution within the solar cell samples, this study's analysis utilized the EM-30AX, a combined Scanning Electron Microscope and Energy Disperse X-ray Spectrometer (SEM-EDS), allowing for a comprehensive investigation of the samples.

### 2.3. Experimental Procedure

#### 2.3.1. Selective Leaching

The leaching solution used in this study to extract silver from the powdered silicon-based solar cells under investigation was nitric acid. The preparation of 1 M, 2 M, and 3 M molar solutions of nitric acid involved a careful dilution process: 64.1 ml of concentrated nitric acid was added to 1000 ml (1 liter) of distilled water, resulting in solutions of the specified molarities. Utilizing an ultrasonic cleaner as brand of Sae–Han Ultrasonic (SH-2340D/40kHz/10l), a mixture of 13 grams of solar cell powder and 100 milliliters of nitric acid solution was subjected to a leaching process at a controlled reaction temperature of 60 °C, where the duration of the ultrasonic mixing was systematically adjusted, ranging from 30 to 120 minutes, to investigate the impact of leaching time on the resulting materials. In this experiment, the concentration of nitric acid and the duration of the leaching process were investigated as the independent variables, while the temperature remained a constant control parameter. Following the leaching process, the resulting solution underwent filtration, with the portion containing silver nitrate utilized in a precipitation reaction to recover the silver; concurrently, the residual portion was washed using distilled water and subsequently dried in an oven at a constant temperature of 100 °C for a full 24-hour period. A method for recovering silver, based on nitric acid leaching and following Equation 1, has been proposed. The dissolution of aluminum and impurities into a nitric acid solution occurs when pulverized solar cells, which include small PV ribbon fragments, are processed; this metal dissolution process adheres to the principles outlined in Equation 2-4.

$$3Ag(s)+4HNO_3(aq) \rightarrow 3AgNO_3(aq)+NO(g)+2H_2O(l) \tag{1}$$

$$2Al(s)+6HNO_3(aq) \rightarrow 2Al(NO_3)_3(aq)+3H_2(g) \tag{2}$$

$$3Cu(s)+8HNO_3(aq) \rightarrow 3Cu(NO_3)_2(aq)+2NO(g)+4H_2O(l) \tag{3}$$

$$3Pb(s)+8HNO_3(aq) \rightarrow 3Pb(NO_3)_2(aq)+2NO(g)+4H_2O(l) \tag{4}$$

#### 2.3.2. Selective Leaching

The process of extracting silver from a solution involves using a reducing agent like copper; the subsequent redox reaction, triggered by placing copper in a silver nitrate solution, is thermodynamically favorable because copper possesses a higher standard reduction potential (+0.34V) than silver (+0.80V). Based on the stoichiometric calculations for the reaction between silver nitrate solution and solid copper (Equation 5), the precise quantity of copper powder required was determined. A vacuum pump was utilized to filter the solid residues remaining after the copper precipitation process; these solids were then dried to a constant weight at 100 °C over a 24-hour timeframe. The chemical composition was determined using X-ray fluorescence (XRF) and inductively coupled plasma optical emission spectrometry (ICP-OES) techniques, and the mineralogical structure was identified using X-ray diffraction (XRD).

$$2AgNO_3(aq) + Cu(s) \rightarrow Cu(NO_3)_2(aq) + 2Ag(s) \tag{5}$$

## 3. Results and Discussion

### 3.1. Silicon Purification Using Selective Leaching

Following the process of nitric acid leaching, the effectiveness of the leaching process was determined by evaluating the grade of silicon purification achieved within the filtered residue that remained after the leaching process. The process of removing the metals from pulverized silicon-based solar cells involves dissolving them in nitric acid, as depicted in the provided equation. Table 2 shows the enhanced silicon purification achieved through leaching experiments, where varying concentrations of nitric acid solution were employed at a consistent temperature of 60 °C for a duration of 60 minutes, resulting in a demonstrable increase in purification. Upon conducting the leaching experiment utilizing a 1 M concentration of nitric acid solution, the silicon content achieved a remarkable purity level of 95.14%. The silicon-based residual contains aluminum and lead as impurities. However, silicon was not detected in the residual, which was thoroughly dissolved into a nitric acid solution. Utilizing a 2 M nitric acid solution resulted in a 98.87% increase in silicon grade, while aluminum and lead content were determined to be 0.85% and 0.28%, respectively. The silicon content reached a 99.17% purity grade using a 3 M concentrated nitric acid solution; conversely, the residue comprised a mere 0.83% aluminum. Analysis of the residuals following leaching with a 1 M concentrate revealed no silver, indicating that the leaching process was ineffective in extracting the silver. Table 2 shows the results of the XRF analysis conducted to ascertain the chemical composition of the samples following the leaching process. When the nitric acid concentration was 3M, the purity of silicon reached above 99%.

**Table 2.**
XRF analysis of the residual powder after leaching the concentrate with nitric acid.

| Concentrate of nitric acid | Element, wt.% | | | |
|---|---|---|---|---|
| | Si | Al | Cu | Pb |
| 1M | 95.14 | 4.34 | - | 0.52 |
| 2M | 98.87 | 0.85 | 0.28 | - |
| 3M | 99.17 | 0.83 | - | - |

The leaching of pulverized solar cells was studied at 60 °C using a 1M concentration to determine how the leaching time impacted the process. Table 3 shows a comprehensive analysis of how the purity of silicon is affected by the leaching process, with results categorized and presented according to the duration of the leaching time. Silicon purification increased to 99.86% with a 30-minute leaching time; however, when the leaching time exceeded 60 minutes, the silicon purification process became less effective and the purity of the silicon decreased. Aluminum constitutes roughly 0.2 percent of the impurity elements found in residual silicon, a material where this abundant element is often found as a contaminant. Based on the observed reaction times, the presence of Si phases was confirmed throughout the leaching process, demonstrating the effective removal of aluminum when the nitric acid leaching exceeded 30 minutes, ultimately resulting in the recovery of silicon with purity exceeding 99%.

**Table 3.**
XRF analysis of residual powder on the leaching durations of 30 to 120 minutes.

| Time, min | Element, wt.% | |
|---|---|---|
| | Si | Al |
| 30 | 99.86 | 0.14 |
| 60 | 99.76 | 0.24 |
| 90 | 99.77 | 0.23 |
| 120 | 99.56 | 0.44 |

The results of X-ray diffraction analysis of the filtered and dried residues obtained after the leaching process, categorized by concentrate type and leaching duration, are presented in Figure 4. Examination revealed the residuals were exclusively silicon phases. Because only a small amount of aluminum is present, no aluminum phases were detected in the residuals. X-ray diffraction (XRD) analysis revealed the presence of a silicon (Si) phase under all experimental conditions tested, encompassing the full range of nitric acid concentrations, as illustrated in the accompanying figure. Confirmation has been received that the concentration of nitric acid increased, resulting in the effective removal of aluminum, copper, and lead.

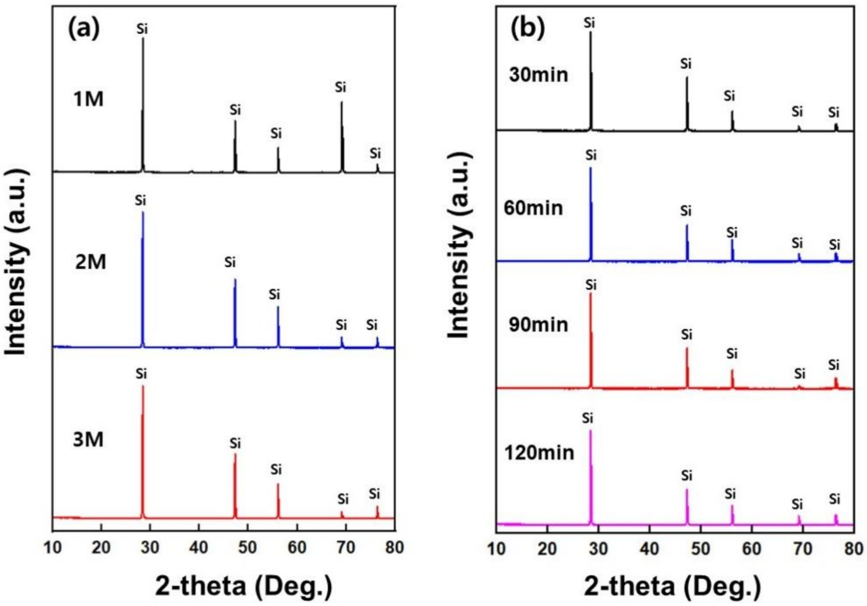

**Figure 4**.
XRD pattern of residual powder after leaching on concentrate (a) and leaching time (b).

## 3.2. Silver Recovery by Precipitation

After the silicon had been purified, an experiment was carried out to recover the silver that remained in solution; this was achieved by using copper in a substitution reaction to precipitate the silver out of solution. In preparation for the precipitation experiment, four samples of the solution were created using a consistent set of parameters—a nitric acid concentration of 3 M, a reaction temperature of 60 °C, and a reaction time of 30 minutes—to ensure uniformity in the samples. ICP-OES analysis of the solution samples was performed to determine the elements present, the results of which are shown in Table 4. Analysis revealed that the solutions contained roughly 30 mg/l of copper and 26 mg/l of silicon, in addition to small, negligible quantities of lead and tin. It is possible that the copper, lead, and tin present in the solution originated from minute fragments of photovoltaic ribbon that were incompletely detached from the solar cells during the manufacturing process. The content of silver present in the solutions ranged from approximately 1.8 to 2.5 mg/l.

**Table 4.**
The chemical composition in the solution after filtration, by ICP-OES.

| **Element, mg/l** | **Sample number** | | | |
|---|---|---|---|---|
| | **1** | **2** | **3** | **4** |
| Cu | 30.224 | 33.941 | 30.228 | 30.552 |
| Pb | 1.544 | 1.609 | 1.354 | 1.376 |
| Sn | 0.5 | 0.6 | 0.4 | 0.6 |
| Al | 11.504 | 13.126 | 10.936 | 11.118 |
| Si | 26.0 | 27.0 | 23.0 | 23.0 |
| Ag | 1.811 | 1.831 | 1.912 | 2.512 |

The solution, upon ICP analysis, was found to contain copper, lead, aluminum, and silver; the stronger reducing properties of silver, in comparison to the other metals detected, were clearly demonstrated by the results. A precipitation experiment was conducted in which 0.25-, 0.5-, 0.75- and 1-gram quantities of copper powder were individually added to separate solutions, each of which was then stirred continuously at a controlled temperature of 60 °C for a period of 30 minutes. Upon the addition of 0.25 g and 0.5 g of copper powder to the solution, no precipitation reaction was observed throughout the precipitation process. The addition of 0.75 grams of copper powder yielded a precipitate weighing 0.23 grams; however, increasing the amount of copper powder to 1.0 gram resulted in a 0.55-gram precipitate. A vacuum pump was used to filter the precipitate, which was then placed in a drying oven at 100 °C for 24 hours to ensure complete dryness. The results of XRF analyses to determine the chemical composition of dried precipitate are shown in Table 5. XRF analysis revealed that when 0.75 g of copper powder was added, the resulting precipitate comprised approximately 49% copper, 48% silver, 1% aluminum, and 0.23% silicon; however, an increase in copper powder to 1 g yielded a precipitate with a significantly different composition of roughly 73% copper, 25% silver, 0.71% aluminum, and 0.17% silicon. Observations revealed a direct correlation between the increased copper addition and a subsequent doubling of the copper concentration within the precipitate; in contrast, the silver concentration in this precipitate showed a decline, reaching a final value of 25.24%.

**Table 5.**
The composition of precipitates, by XRF.

| **Cu addition, g** | **Element, %** | | | |
|---|---|---|---|---|
| | **Cu** | **Ag** | **Al** | **Si** |
| 0.75 | 49.9 | 48.65 | 1.23 | 0.23 |
| 1.0 | 73.88 | 25.24 | 0.71 | 0.17 |

Figure 5 presents the findings from X-ray diffraction (XRD) analyses conducted to determine the precise compositional makeup of the dried precipitate sample. Upon performing XRD analysis on precipitates recovered after adding 0.75 g and 1 g of copper, silver was identified as the dominant phase, while further analysis using XRF detected the presence of copper and copper oxide as impurities.

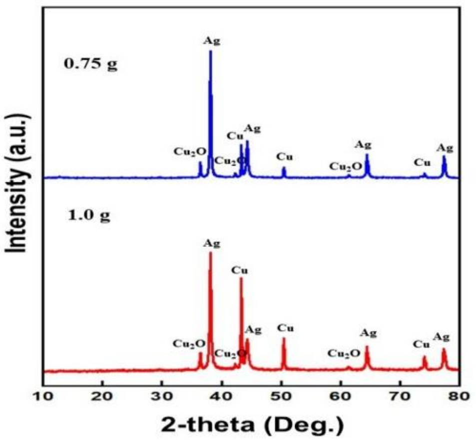

**Figure 5.**
XRD pattern of precipitates with addition of copper at 0.75 g and 1.0 g.

Figure 6 illustrates the recovery rates achieved for silver through chemical precipitation, utilizing copper additions of 0.75 grams and 1.0 gram respectively, from the filtered solution remaining after the selective leaching process had been completed. While the addition of copper resulted in an elevated copper concentration in the resulting precipitate, a silver recovery rate of 93.3% was successfully achieved when the maximum amount of copper added was 1.0 g.

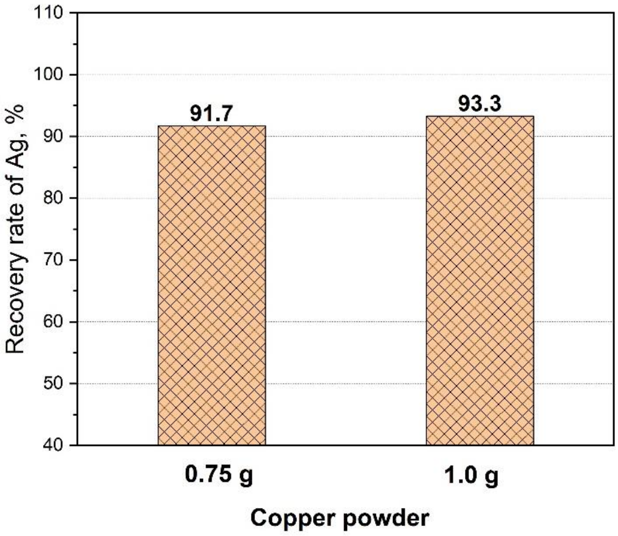

**Figure 6.**
Recovery rate of silver by copper precipitation.

## 4. Conclusions

The purpose of this investigation was to utilize nitric acid (HNO3) solution leaching and subsequent precipitation methods to effectively recover silicon and silver, two valuable metals, from pulverized solar cell materials. The recovery of silicon and silver was investigated using nitric acid leaching; concentrates were prepared with molar solutions of 1 M, 2 M, and 3 M and subjected to reaction times ranging from 30 minutes to 120 minutes at a constant temperature of 60 °C to determine the effectiveness of this recovery method. The purification process, involving a nitric acid solution, achieved a silicon purity of over 99% from the pulverized solar cell; this was accomplished by using a 3 M nitric acid concentration, which successfully removed copper, lead, and other impurities completely. Furthermore, experiments revealed that a leaching period of 30 minutes proved adequate for purifying silicon to a level greater than 99%, a result that was shown to be dependent on the duration of the leaching. During the silver recovery process, employing a precipitation reaction from solution, the addition of 0.25 grams and 0.5 grams of copper powder yielded no products; however, a precipitate was successfully formed upon the subsequent addition of 0.75 grams and 1 gram of copper. Following the addition of 0.75 g and 1 g of copper, the recovered precipitates contained approximately 48 wt.% and 25 wt.% silver, respectively; this decrease in the silver content of the precipitate corresponded to an increase in the amount of copper added. During precipitation, the addition of copper causes aluminum to deposit in the silicon residue; this is a consequence of aluminum's lower reducibility compared to silver and copper. The results of this study demonstrate the successful recovery of silicon to a purity exceeding 99%, alongside the recovery of a silver-containing precipitate; these achievements are expected to yield significant economic and environmental advantages, namely a reduction in the raw materials required for solar module manufacturing and a decrease in environmental contamination originating from waste disposal sites.

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