PT RECOVERY FOR END-OF-LIFE HALIDE PEROVSKITE PHOTOELECTROCHEMICAL DEVICES

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Abstract

Perovskites refer to broad classes of materials that share the same crystal structure as the calcium titanium oxide mineral with the chemical formula of $CaTiO_3$. A Pt-based halide perovskite is a vacancy-ordered double perovskite. It typically has the formula A_2PtX_6, where A is a monovalent cation (Cs) and X is a halide (Cl, Br, I), and alternate B cation sites are intentionally left vacant, making the unit cell as A_2PtX_6. In this 0D or vacancy-ordered structure, Pt exists in 4+ state and the perovskite phase is environmentally stable and is free of the toxic PbI_2. Since Pt exhibits chemical stability, high electrical conductivity, and catalytic properties, it can enhance the properties of perovskite phase. Pt-based halide perovskites are generally studied for their potential in solar cells, thermoelectric devices, and water splitting photocatalysis. Pt is typically very expensive and scarce due to its complex properties, making it a critical metal. Extracting platinum from Pt-based halide perovskite thin-film semiconductors has significant importance for the circular economy and for other applications that require Pt-based technologies, which allows for sustainability, cost efficiency, and material optimization. This study focuses on using a chloride and nitrate based aqua regia along with ammonium chloride to extract $(NH_4)_2PtCl_6$,from Pt-based halide perovskite thin-film semiconductors, which will then be used to extract elemental platinum in the future. These thin films are placed in the aqua regia, and then ammonium chloride is added to form a precipitate. This precipitate, $(NH_4)_2PtCl_6$, will then be heated in the future so that elemental platinum powder can be produced. To evaluate the purity of the $(NH_4)_2PtCl_6$ powder extracted, X-ray Fluorescence (XRF) will be used which measures emitted X-rays to determine chemical composition. This method will be used to analyze the purity of the recovered $(NH_4)_2PtCl_6$ powder. In the future, elemental Pt will also be analyzed once extracted, which can then be used as a catalyst in fuel cells or as catalyst in the automotive industry to convert harmful exhaust gases to less harmful emissions.

Keywords: Pb-free, Pt-based halide perovskites, extraction, cost efficiency, circular economy

1. Introduction and Motivation

Global cumulative deployment of photovoltaic (PV) modules is projected to increase to 4.5 TW by the end of 2050, with a large mass of modules to be discarded at the end-of-life (EOL).[1] Responsible life cycle management of PV systems is crucial not only for complying with regulations but also for improving project economics and the levelized cost of electricity (LCOE) of a given PV project by using cost-effective, high-value recycling technologies. Even though the mass of semiconductor materials is less than 1% of the module mass for thin-film photovoltaics, by managing the disposal of end-of-life (EOL) PV modules and components, asset owners can avoid potential environmental liabilities and negative impacts on their reputation. Current recycling of PV modules (and electronics waste in general) entails an initial stage of energy-intensive shredding and physical separation of metal streams, semiconductor materials and non-metallic fractions followed by pyrometallurgical or hydrometallurgical leaching and metal recovery[2-3]. Physical separation methods are common across PV technologies recovering glass, aluminum frames and interconnects; while only small fractions of Ag, Te and critical metals are recovered due to trace concentrations. Hydrometallurgical leaching methods common in the microelectronics industry such as acid and cyanide offer efficient mineral recovery, however, pose toxicity and environmental threats leading to adoption of greener leaching technologies such as alkaline solvents, chelating agents, or bioleaching.[4] To enhance the leaching reaction kinetics with greener solvents; chemical-mechanical treatments like ultrasonic, supercritical-CO_2, oxidizing agents, etc. have been used in recovery of electronics waste.[5-6] In this paper, we review the state-of-art for recycling of halide perovskite solar cells and discuss our recent results on extraction of critical metals from Pt-halide perovskites, a promising Pb-free composition for PV, light emitting diodes, thermoelectric devices and H_2 production.

2. Review of Related Work

2.1. Recycling of Halide Perovskites

Halide perovskite solar cells (HPSCs) have shown significant promise for next-generation solar cells due to their high-power conversion efficiency, low manufacturing costs and tunable properties. The presence of Pb in HPSCs poses environmental challenges due to degradation, potential leaching of Pb and EOL disposal. Therefore, recycling and recovery technologies are critical to minimizing the environmental footprint and complying with regulations such as the Waste Electrical and Electronic Equipment Directive.[7-8] The literature in recycling of HPSCs can primarily be divided into two categories, studies that have focused on recovery of expensive transparent conductive oxides (TCOs)

to reuse for device re-fabrication at EOL of the degraded perovskite layers; and other studies that also recover the spent Pb.[9] Pb-recovery is achieved by the use of butylamine solvent,[10] deep eutectic solvents (DES)[11], and zeolite based adsorbents.[12] Methods like DES or NH_3:H_2O[13] treatment can recover up to 99.8% Pb or PbI_2 and reduces the toxicity risks of HPSCs, however, further research is needed to obtain high purity precursors for reuse. Recycling of expensive TCOs offers economic benefits, and recycling of Pb offers environmental benefits; but system level recycling with selective recovery has not been demonstrated and is important for industrial integration and commercialization.[14]

2.2. Pt halide perovskites

Platinum (Pt) halide perovskites have emerged as a promising alternative to lead (Pb) halide perovskites, which are extensively studied and widely applied in photovoltaic technologies.[15-20] Pt halide perovskites exhibit exceptional electronic properties, including a high absorption coefficient across a broad wavelength range, a bandgap of approximately 1.4 eV that is ideal for photovoltaic applications (optimal bandgap of 1.3-1.4 eV according to Shockley-Queisser limit),[21] and a carrier lifetime exceeding 2 μs.[15] Pt halide perovskites not only mitigate the concerns about the Pb toxicity in Pb-based perovskite but also address the issue of stability. Among Pt-based perovskites, the vacancy-ordered double perovskite Cs_2PtX_6 (X=I, Br, Cl) stands out due to its remarkable stability, even in acidic and basic solutions, making it a strong candidate for photocatalytic applications.[16-18]

However, the high cost of Pt remains a significant challenge for the widespread adoption of these materials. While strategies such as elemental substitution (e.g., Ni-Pt mixing) have been explored to lower costs, an alternative approach involves the extraction and recycling of Pt from end-of-life Pt-based perovskite materials.[22] This recycling strategy offers a practical solution to address the economic barrier associated with Pt halide perovskites.

2.3. Pt Extraction

Three different methods were analyzed before choosing which approach to utilize. The first method focused on using amine based extractants to extract elemental platinum.[23] With this method, a tertiary amine compound modeled by the general chemical formula R_3N would be used as the main chemical. One example of a compound that could be used is tri-n-octylamine ($C_{24}H_{51}N$). This process includes 4 general steps: the leaching of platinum with HCl, the extraction of the solvent using the amine, the stripping of Pt using HCl as the agent, and the reduction of Pt using Zn powder (could be replaced by Fe, Mg, Al, Cu). However, since amine-based extractants require very careful handling due to toxicity and may co-extract other metal ions like Cs which would lower purity Pt recovery, this was not a preferred method. The second method focused on using nitrates to extract elemental platinum.[24] With this method, a nitrate like ammonia (NH_3) would be used as the main chemical. This process involves the formation of a soluble complex using HCl and then a complex with ammonia and HNO_3 to form a precipitate. With the use of H_2 as the stripping agent, elemental Pt can be extracted. However, using purely HNO_3 would produce many nitroso-compounds and possibly other unwanted byproducts which could be harmful. Additionally, the usage of H_2 gas can be hazardous if not handled properly. For these reasons, this method was also not preferred. The last method focused on using chlorides to extract elemental platinum.[25] With this method, a chloride like ammonium chloride (NH_4Cl) would be used as the main chemical. This process is similar to the second method with the formation of a soluble complex and then a complex with the main chemical, ammonium chloride. However, instead of using a stripping agent, heating of the precipitate can be used to extract the elemental platinum. However, a chloride based extractant may not allow for high purity Pt recovery due to other metal compounds (like Cs) forming similar complexes, so this method was also not preferred. Considering these limitations, a modified method was applied, combining the second and third methods. This modified method improves upon the previously discussed methods because it eliminates the need for H_2 gas, uses NH_4Cl solution as to form a complex which is more stable than amines and HNO_3, and allows for higher purity Pt recovery. By addressing most of the limitations of the previously discussed methods, the modified method allows for a safer and more effective way to extract Pt.

2. Technology Approach

2.1. Materials

The Pt perovskite thin films used for Pt extraction and recycling in this study are obtained from previous fabrication experiments conducted in our lab. The composition of these Pt perovskite thin films are $CsPtX_3$ (DMSO),

Cs_2PtX_6, and Cs_2PtX_6/Pt (X = I, Br, Cl), respectively. Nitric acid (HNO_3), hydrochloric acid (HCl), and ammonium chloride (NH_4Cl) are obtained from Sigma Aldrich (USA).

2.2. Methods

We first prepared the aqua regia solution for platinum ion leaching. 25 mL concentrated nitric acid (HNO_3) was slowly transferred into 75 mL concentrated hydrochloric acid (HCl) with thorough mixing until the solution is in bright orange color (**Figure 1a**).[2] Following this, the perovskite thin films (**Figure 1b**) containing Pt^0, Pt^{2+}, and Pt^{4+} were scraped and leached into soluble platinum salts by dissolving in the aqua regia solution. A pipette was applied to facilitate this process (**Figure 1c**). Once the thin films were completely dissolved, the solution was set on a hot plate over night with stirring allowing the formation of H_2PtCl_6 and condensed solution for following procedure. The reactions can be described in the following chemical reaction equations (1-3):

$$3Pt + 4HNO_3 + 18HCl \rightarrow 3H_2PtCl_6 + 4NO\uparrow + 8H_2O \quad (1)$$

$$PtCl_2 + 2HNO_3 + 4HCl \rightarrow H_2PtCl_6 + 2NO_2\uparrow + 2H_2O \quad (2)$$

$$PtCl_4 + 2HCl \rightarrow H_2PtCl_6 \quad (3)$$

After that, NH_4Cl solution (1 mol/L) was prepared by dissolving 5.349 grams of ammonium chloride into 100 mL of distilled water. Dilute HCl was added to the H_2PtCl_6 solution to get rid of any nitroso compounds (NO, NO_2) that were present. After this, the solution was cooled to 50°C, the NH_4Cl solution was added to the condensed H_2PtCl_6 solutions, 10 mL at a time, forming ammonium chloroplatinate precipitant (**Figure 1e**)[26]. The reaction in this step is as following.

$$H_2PtCl_6 + 2NH_4Cl \rightarrow (NH_4)_2PtCl_6\downarrow + 2HCl \quad (4)$$

The ammonium chloroplatinate was then separated from the solution using a syringe strainer (25mm diameter and 0.22um pore size) (**Figure 1f**). The resultant solution was clear in light yellow, and the powder was air dried and collected (**Figure 1g**). The clear solution in Figure 1g is mainly made of high concentration of Cs+, which shows light yellow color.[27]

X-ray fluorescence (XRF) characterization measurement was conducted on Horiba X-Ray Analytical Microscope XGT-9000. The 15 micro ultra-high intensity probe was applied. The powder sample was sandwiched between two X-ray-transparent plastic films.

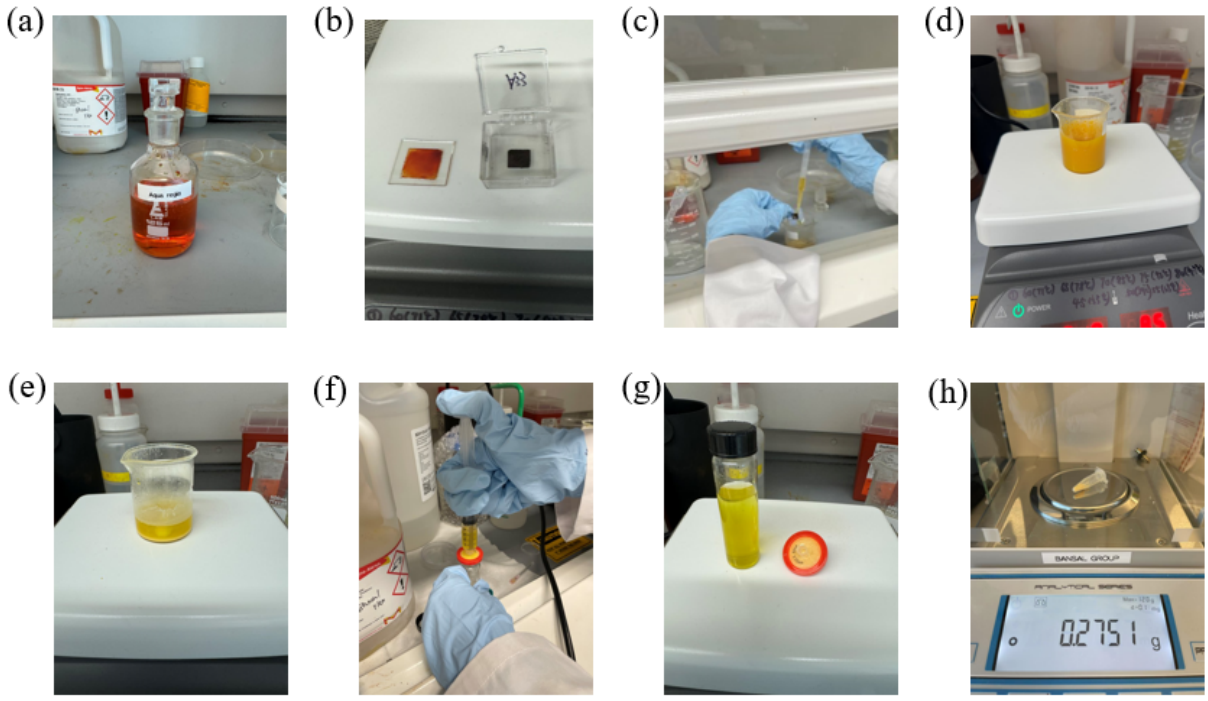


Figure 1. *The procedure of Pt extraction from Pt halide perovskite thin films. (a) Fresh aqua regia solution. (b) The Pt perovskite thin films. The orange thin film in the figure is* $CsPtI_3(DMSO)$ *and the black thin film is* Cs_2PtI_6. *(c) Dissolving the Pt halide perovskite in aqua regia (d). Condensing the solution and adding HCl to remove* NO_x. *(e) Precipitation after the addition of* NH_4Cl *solution. (f) Separate the precipitate from solution. (g) The resultant clear solution after the separation and the powder collected. (h) The collected powder with weight of 0.2751g.*

3. Results and Discussion

Aqua regia solution was applied for leaching the platinum ions in the Pt based perovskite thin films, as detailed described in the Method section. Then, the leached orange solution was set on hot plate for reaction to get condensed solution containing H_2PtCl_6. After that, the NH_4Cl solution was added at lower temperature as extractants. During the process, the Pt ions were extracted in the form of precipitate $(NH_4)_2PtCl_6$, resulting in the light orange powder. 0.275 g powder was collected in this process, as shown in Figure 1h. The light-yellow clear solution should be composed of Cs^+ ions, acids, and possible tiny size $(NH_4)_2PtCl_6$ particles, etc.

The collected light-orange powder was then characterized by XRF. The high-energy primary X-ray beam excited the sample by ejecting the inner-shell electrons of the elements in the sample. To fill the vacancy, the electron from a higher-energy outer shell of these elements drops down to the lower-energy inner shell and emits characteristic secondary (fluorescent) X-rays. Detection of these fluorescent X-rays enables us to have more elemental details of the sample. We sandwiched the collected light-orange powder sample between the X-ray-transparent thin film and placed it in the sample chamber (**Figure 2a**). The sample was then analyzed in 4 different areas, as shown in **Figure 2b**, creating 4 different spectrums of the chemical composition (**Figures 2c-f**). It can be found that the Pt peak intensity is most abundant, suggesting the successful extraction of the Pt. However, the Cs and partial halide like Br and I still exist in the powder. This impurity should result from the lack of multiple-step-repurification (we applied one-step instead) and the Cs halide salt shows low solubility in aqueous solution. The concentrations of each element in the powder are shown in **Figure 3**. However, nitrogen was excluded because the film paper used in the XRF was mainly composed of nitrogen compounds (99.9987% concentration).

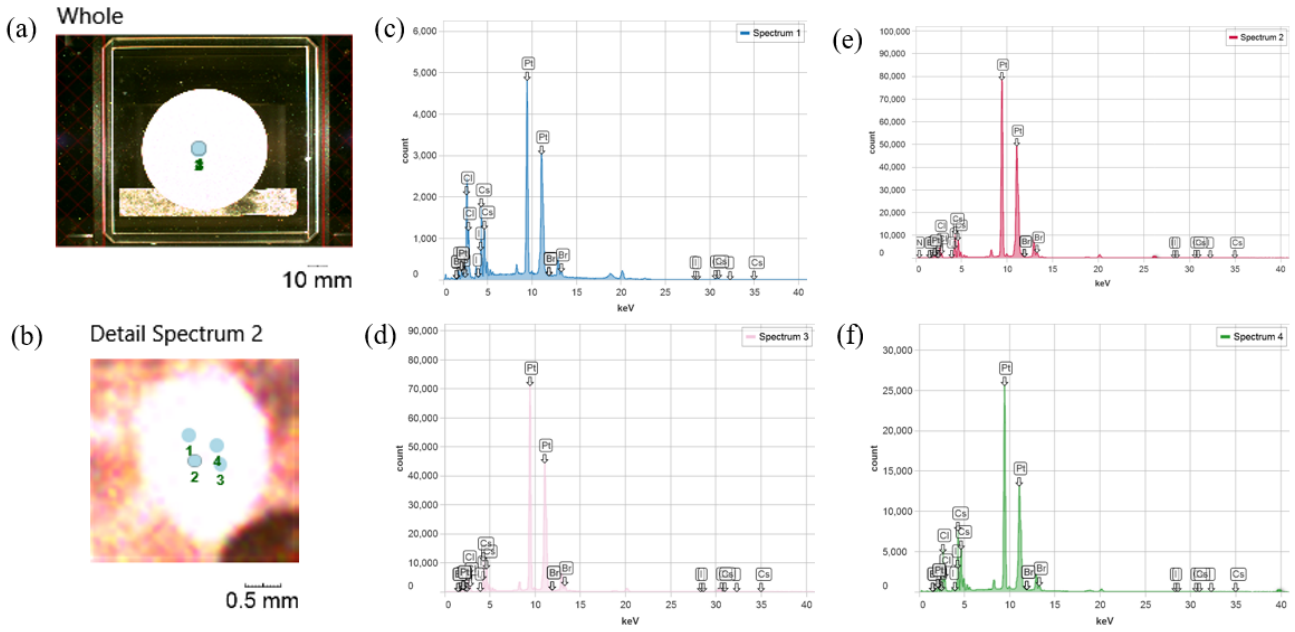
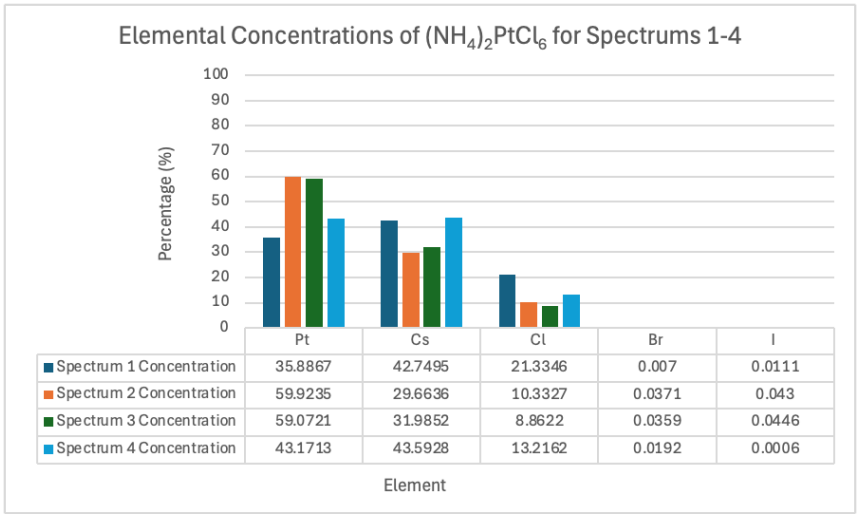


Figure 2. *(a) Sample powder in the XRF chamber. (b) 4 different areas of the powder that were analyzed. (c) Spectrum 1 chemical composition of the powder. (d) Spectrum 3 chemical composition of the powder. (e) Spectrum 2 chemical composition of the powder. (f) Spectrum 4 chemical composition of the powder.*



	Pt	Cs	Cl	Br	I
Spectrum 1 Concentration	35.8867	42.7495	21.3346	0.007	0.0111
Spectrum 2 Concentration	59.9235	29.6636	10.3327	0.0371	0.043
Spectrum 3 Concentration	59.0721	31.9852	8.8622	0.0359	0.0446
Spectrum 4 Concentration	43.1713	43.5928	13.2162	0.0192	0.0006

Figure 3. *Elemental Concentrations of (*$(NH_4)_2PtCl_6$ for Spectrums 1-4

One step of repurification was completed to increase the purity of the $(NH_4)_2PtCl_6$ salt collected. This repurification step involved mixing the $(NH_4)_2PtCl_6$ salt in approximately 50 mL of distilled water. The addition of the distilled water to the salt allowed for the compound CsCl to readily dissolve, leaving the insoluble $(NH_4)_2PtCl_6$ powder in the solution. The solution was then mixed with the vortex mixer for 5 minutes. Following this, the powder was extracted using a syringe strainer (25mm diameter and 0.22um pore size). Once this was completed, the sample powder was analyzed with the XRF machine, however, there were minimal differences between the previous results, indicating that CsCl wasn't the only compound that was formed, but instead, another unknown Cs complex. These results are shown in **Figure 4** and **Figure 5**.

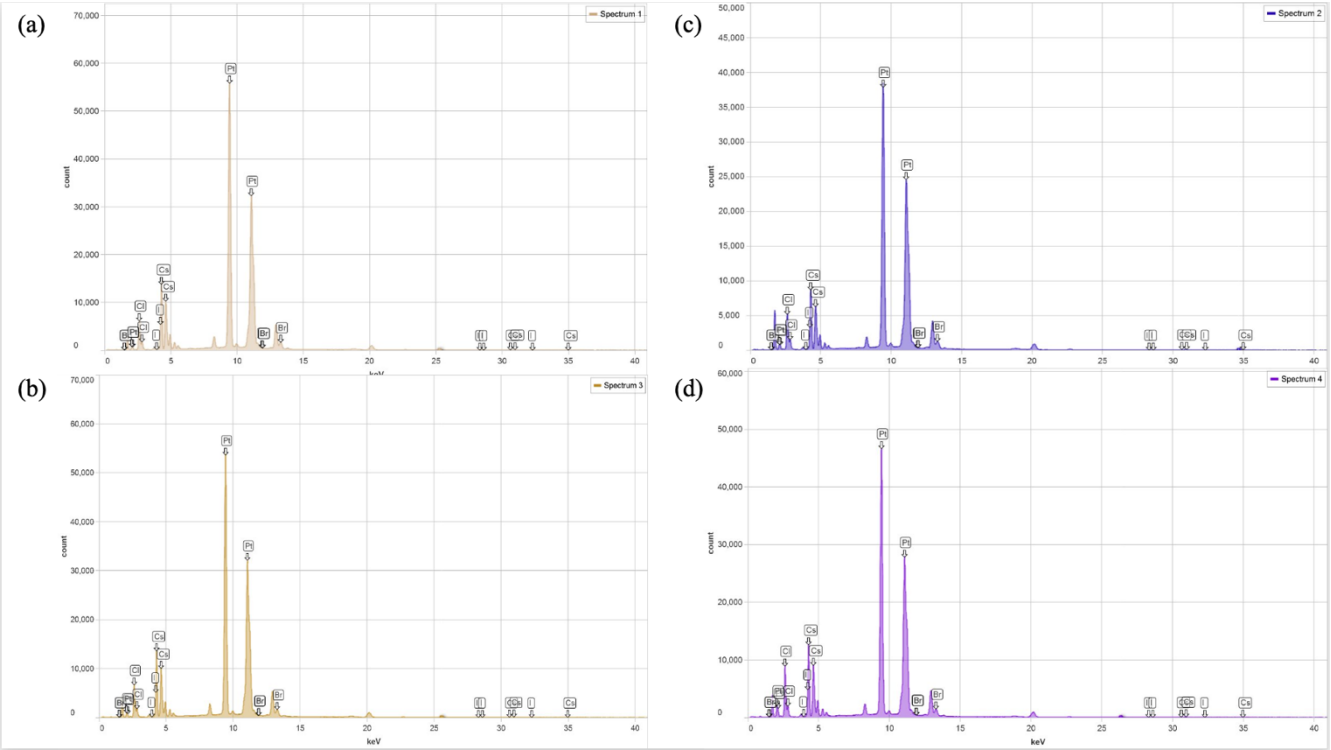
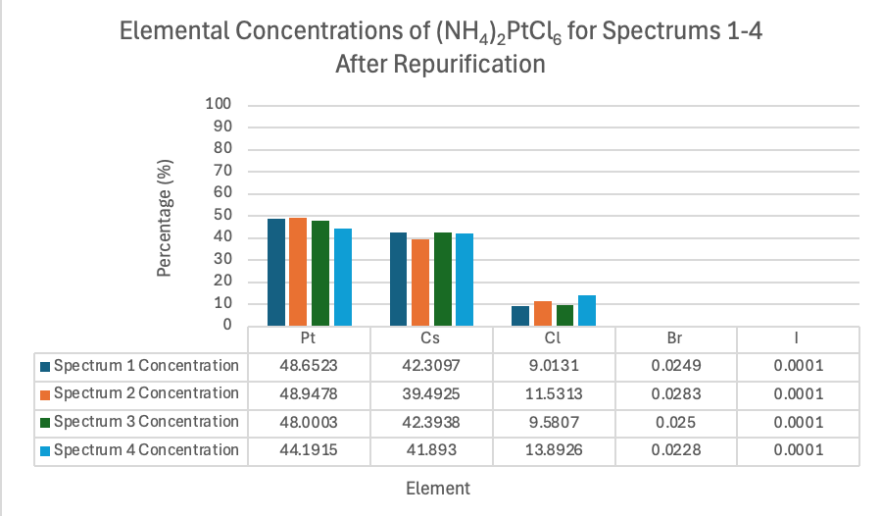


Figure 4. *(a) Spectrum 1 chemical composition of the powder after repurification. (b) Spectrum 3 chemical composition of the powder after repurification. (c) Spectrum 2 chemical composition of the powder after repurification. (d) Spectrum 4 chemical composition of the powder after repurification.*



	Pt	Cs	Cl	Br	I
Spectrum 1 Concentration	48.6523	42.3097	9.0131	0.0249	0.0001
Spectrum 2 Concentration	48.9478	39.4925	11.5313	0.0283	0.0001
Spectrum 3 Concentration	48.0003	42.3938	9.5807	0.025	0.0001
Spectrum 4 Concentration	44.1915	41.893	13.8926	0.0228	0.0001

Figure 5. *Elemental Concentrations of* $(NH_4)_2PtCl_6$ for Spectrums 1-4 After Repurification

In our future work, we will continue to conduct multi-step repurification using a different method to get rid of any excess Cs. This method involves remixing the sample powder with the aqua regia and allowing the solution to condense overnight. After condensing, the NH_4Cl solution will be added again to form the complex $(NH_4)_2PtCl_6$. Additionally, we will characterize the salt with Inductively Coupled Plasma Mass Spectrometry (ICP-MS) which focuses on analyzing the sample based on the ionization of elements once the sample is digested and turned into liquid. This will also indicate how much Pt remains un-extracted in the solution. Moreover, in this study we set

$(NH_4)_2PtCl_6$ salt as the final product and in the future work we will further transfer the salt into elemental Pt by reduction or high temperature thermal treatment, as shown in the following equation:

$$3(NH_4)_2PtCl_6 = 3Pt + 16HCl + 2NH_4Cl + 2N_2\uparrow \quad (5)$$

4. Conclusions and Recommendations

For this research, a HCl/HNO_3 aqua regia and ammonium chloride salt (NH_4Cl) was used to precipitate out an ammonium chloroplatinate ($(NH_4)_2PtCl_6$) powder. This powder sample was then analyzed with an XRF machine, which concluded that there were mostly Pt and Cs ions in the sample, which indicated that there were some impurities that existed due to some Cs ions from the thin film remaining in the powder. This can easily be reduced with multi-step repurification. In the future, after most of the Cs ions are removed from the sample with multi-step repurification, the Pt will be reduced further to its elemental form, and the XRF machine will be used again to analyze any impurities. Based on the conducted research, this method worked well in isolating the salt powder from the solution, however, some improvements could be made with multi-step repurification to get pure $(NH_4)_2PtCl_6$, so that pure elemental Pt can be extracted in the future.

After conducting the multi-step repurification and the extraction of the elemental Pt, the resultant Pt can be reused and recycled for other purposes, such as a catalyst for fuel cells (water splitting), as a catalyst in the automotive industry to convert harmful exhaust to less harmful emissions, solar cells, and thermoelectric devices. The scarcity and cost of Pt places emphasis on the importance of the circular economy and recycling Pt for applications that require it. As Pt is defined as a critical metal, it is essential to be able to isolate it from compounds to allow for sustainability, cost efficiency, and material optimization.

Acknowledgements

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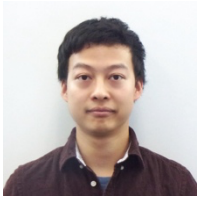

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