

Recovery of Ammonium Perchlorate from Obsolete Ammunition and Its Application in Synthesis of Lithium Perchlorate

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Abstract: Ammonium perchlorate (AP) is used as a strong solid oxidizer in propellants, gun powder, fireworks, and light signals. Recycling or recovery of AP from demilitarization or outdated propellant items is a challenging task. Herein, we report the purchase of recycled crude AP from commercially, and then subsequently AP was purified through filtration with activated carbon and recrystallization. After recrystallization, the yield of AP was 85%, and the resultant product was characterized by various techniques including Fourier-transform infrared spectroscopy, nu-

clear magnetic resonance spectroscopy, X-ray diffraction, scanning electron microscopy, and inductively coupled plasma mass spectrometry. These results indicate that recrystallized AP had excellent purity and crystallinity compared to crude AP. In addition, purified AP was used as source material for the synthesis of lithium perchlorate (LP), which was characterized using various analytical techniques. This process provides a new avenue for recycled AP that could be effectively used as starting material for synthesis of LP.

Keywords: Recycling inorganic salt • Ammonium perchlorate • Lithium perchlorate • Obsolete ammunition

1 Introduction

Demilitarization is a neutralization of obsolete ammunition, typically performed by incineration. During the incineration process, the release of large amounts of energy with gases of CO₂, HCl, and other particles such as aluminum monoxide (AlO), alumina (Al₂O₃), which create environmental pollutions [1–3]. On the other hand, the primary explosive residual of perchlorate salts is highly soluble in water and higher density than water, excess of perchlorate salt in the human body inhibit thyroid production [4–7]. Therefore, many companies and military departments ceased burning obsolete ammunition and have stored it in bunkers until the development of a safe and new method. To address this issue, the demilitarization business and related research are currently emerging [7–9]. Ammonium perchlorate is an important strong solid oxidizer that has diverse applications in the propellant industry, fireworks, and satellites. In addition, as AP generates oxygen and other gases when decomposed, it is used in airbags, ejection seats, and construction-destruction industries [10]. AP is stable under normal temperature and pressure, but when decomposition due to burning can produce a high-pressure explosion. Although large amounts of the AP compound are used in the aerospace launch industry and military application, it is one of the best propulsion materials [11–12]. However, as the propellant becomes superannuated, the propulsion system is no longer controlled. Hence, obsolete propulsion motors have been discarded without consideration of repurposing

AP. Instead of incineration of propellant in open space, recently many researchers have focused on the recycling of AP by using the suitable method; however, this is a challenging task due to environmental safety, purity, and the high cost of processes [13–15]. Several methods are dealing with recycles of AP from solid propellant [16–17], among them, leaching of AP into aqueous solution by using water

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is widely used method [15]. Marraud et al. invented the biological purification [18] of ammonium perchlorate from aqueous solution using nitrification/denitrification sequence with the anaerobic condition and a similar method was also reported by others. [18–20]. Recently, our research group successfully separated AP from propellant mixtures via an automated, self-contained, and environmentally responsible process [7]. The recycling program, which demilitarizes more than 100 units of 130 mm propellant motors per day, has proven to be cost-effective. The purity of the recycled AP was approximately 80% and other presumed impurities were mainly water and polymer-related residues [21]. Hence, it is necessary to develop a suitable purification method to get a high purity of AP from the recycling process. We describe a new strategy for purification of AP from obsolete propellant motors by three processes: (i) recrystallization, (ii) filtration, and (iii) recrystallizations. The purified AP is being used as starting materials for the synthesis of lithium perchlorate (LP) derivatives. Generally, LP is used in lithium ion polymer batteries as an electrolyte in a gel-type mixture with propylene carbonate and dimethoxyethane [22–25]. This project could have great significance in demilitarization research because transformation of obsolete ammunition into energy-related materials has not previously been realized.

Herein, we first purified recycled AP through recrystallization and filtration with activated carbon and a membrane filter. An 85% yield was obtained after recrystallization of crude AP, and the resultant product was characterized by various techniques including Fourier-transform infrared spectroscopy, nuclear magnetic resonance spectroscopy, X-ray diffraction, morphology analysis, and inductively coupled plasma mass spectrometry. In addition, purified AP was used as source material for the synthesis of lithium perchlorate, which was characterized using various analytical techniques..

2 Experimental Section

2.1 Materials

$\text{LiOH}\cdot\text{H}_2\text{O}$, Li_2CO_3 , and all solvents used were purchased from Kanto Chemical with HPLC grade. Recycled AP was obtained from JJPROTECH (South Korea) after an automated recycling process and was analyzed via the MIL-A-23442A method. The micro-filters used included cellulose nitrate membrane filters (Macherey Nagel: Dia. 47 mm, pore size 0.45 μm) or GF/C glass microfiber filters (Whatman: Dia. 47 mm, pore size 1.2 μm).

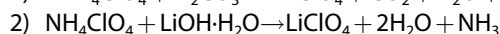
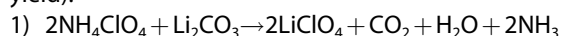
2.2 Purification of AP

The purity of the recycled AP was analyzed systematically with MIL-Spec. The MIL-Spec results revealed that the purity

of recycled AP was $\sim 85\%$. Recycled AP was further purified by recrystallization and filtration with activated carbon and a membrane filter. Initially, 200 g of crude AP (AP-0) was dissolved in 1 L of hot distilled water, and the solution was filtered through 10 g of activated carbon (AP-A). Another 10 g of activated carbon was mixed with the filtrate and stirred for 1 h, followed (AP-B) by filtration through a membrane filter (AP-C). The filtrate was cooled, and AP was recrystallized at 4 $^{\circ}\text{C}$ overnight to produce white crystalline material recovered in $\sim 85\%$ yield as shown in figure 1.

2.3 Synthesis of LP from Purified AP

To prepare LP from purified AP, we tested two synthetic pathways using different Li compounds and are given as follow. First, 10 g of AP was mixed with different Li compounds (Li_2CO_3 and $\text{LiOH}\cdot\text{H}_2\text{O}$) in a molar ratio of 1:2 in 20 mL of distilled water and stirred vigorously at 100 $^{\circ}\text{C}$ for overnight. The solution was cooled to room temperature and held overnight at 4 $^{\circ}\text{C}$. The obtained white crystalline material was washed three times with hexane: Isopropyl alcohol (10:1, v/v) and dried in a vacuum oven at 80 $^{\circ}\text{C}$ (90% yield).



2.4 Material Characterization

Fourier transform infrared spectra of the purified samples were collected using the Nicolet Impact 400 FT-IR, USA, with the KBr disk method. The ^1H NMR data of the purified AP and LP in $\text{DMSO}-d_6$ were recorded on a Bruker 400 MHz NMR spectrometer. Structural analysis of the purified samples was performed by using an X-ray diffractometer (XRD,

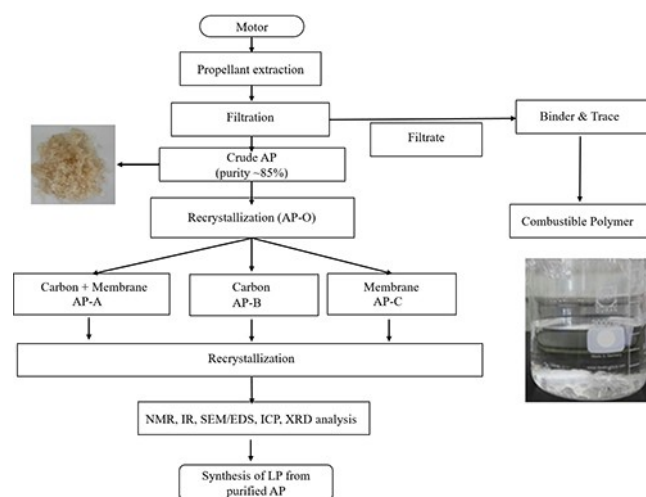


Figure 1. Schematic illustration that purification of crude AP.

X'pert-MRD, Pro Philips) with CuK alpha radiation ($\lambda = 1.54178 \text{ \AA}$). Morphology analysis of purified samples was carried out using a field emission scanning electron microscope (FE-SEM) with JSM-6400 (JEOL, Japan). Inductively coupled plasma (ICP) analysis was performed with the ICP-MS X series II (Thermo Scientific, USA) to analyze all inorganic impurities. Impurities such as Ca, K, and Na were measured with the EPA 3050B or 6010 C method.

3 Results and Discussion

FT-IR spectra confirmed the chemical structures of purified AP and synthesized LP. Figure 2a shows the FT-IR spectra of the compounds before and after purification. Characteristic peaks that appeared at $3250\text{--}3700 \text{ cm}^{-1}$ and 1403 cm^{-1} belong to stretching and bending vibrations of ammonia, respectively [26]. A peak located at 627 cm^{-1} corresponds to stretching vibrations of the ClO_4^- coordinate covalent bonds, while a broad peak around 1090 cm^{-1} is from the asymmetric stretching vibration of ClO_4^- [27]. Before purification, we observed multiple peaks resulting from impurities (AP-O), but after purification, only broad ammonia bands were detected, which indicated the removal of bulk impurities. The ^1H NMR spectra for the compound before

and after purification are shown in Figure 2b. From the spectra, we confirmed the absence of organic carbohydrate impurity. The peaks at 2.49 ppm and 3.33 ppm correspond to DMSO-d_6 solvent and water respectively. The splitting of the proton signal in the NMR at 7.11 ppm was due to the coupling with ^{14}N ($\text{Spin} = 1$), it is observed when ammonium ion is in an acidic aqueous solution. Further, purification of the samples, the multiple peaks in the NMR at 7.11 ppm were becoming more gentle and singlet which indicates the high purity of the samples (AP-A, AP-B, and AP-C) [28], which was consistent with the IR spectra results. These results confirmed the improved purity of AP after recrystallization of crude AP. Figure 3a shows FT-IR spectra of synthesized LP using purified AP and LP precursors such as Li_2CO_3 and $\text{LiOH}\cdot\text{H}_2\text{O}$. We observed loss of the absorption peak corresponding to the N-H bending vibration at 1403 cm^{-1} of AP and the appearance of a new peak corresponding to the stretching vibration of LiClO_4^- at 1633 cm^{-1} (Figures 2a and 3a). Peaks at 627 cm^{-1} and 1090 cm^{-1} corresponding to the stretching vibrations of ClO_4^- were observed in the perchlorate compounds [29], which confirmed the formation of LP from purified AP.

Further, to obtain more accurate information on the synthesized LP by the aforementioned methods, ^1H NMR spectra of each product were analyzed and compared to observe the presence of additional hydrogen peaks from organic impurities. The solvent used for analysis was DMSO-d_6 , and the 400 MHz ^1H NMR spectra are shown in Figure 3(b–c). First, the DMSO-d_6 solvent peak in Figure 3(b–c), which presents the ^1H NMR spectrum for material presumed to be LP synthesized with Li_2CO_3 and $\text{LiOH}\cdot\text{H}_2\text{O}$, appears at 2.49 ppm, and a water peak is observed at 3.3 ppm due to moisture contained in the prepared NMR sample. Figure 3(b–c) shows no peaks (no hydrogen bonds) except those of solvent and water, indicating the absence of impurities that contain hydrogen bonds or remaining AP.

The morphology of purified AP and synthesized LP was investigated through FE-SEM analysis as shown in Figures 4 and 5. Figure 4 a–b shows FE-SEM images of purified AP-A and AP-C that indicate the presence of a few micrometers of purified AP, and energy dispersive x-ray analysis (EDX) shows AP-C has improved purity compared to AP-O, AP-A, and AP-B, as shown in Figure 4c. The surface structure of synthesized LP was observed by FE-SEM, as shown in Figure 5a–b. FE-SEM images of LP from Li_2CO_3 and $\text{LiOH}\cdot\text{H}_2\text{O}$ displayed rod and aggregated granular morphology with amorphous nature. We also determined the degree of purity using ICP-MS analysis for purified AP and synthesized LP, with the results shown in Table 1–3. The purity of the initial sample (AP-O) was about 85%, which increased to higher than 99% after treatment as shown in Table 2. Among the 12 impurity elements analyzed, most of the impurities were removed and negligible amounts of Ca, Na, and K exist in the samples as shown in Table 1. We conclude successful filtering and recrystallization for increasing AP purity. Nevertheless, the second recrystallization of a sam-

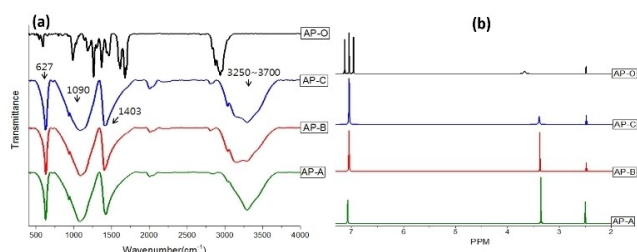


Figure 2. FT-IR spectrum of (a) IR spectra of AP from the purification step; (b) ^1H NMR spectra of AP from the purification step.

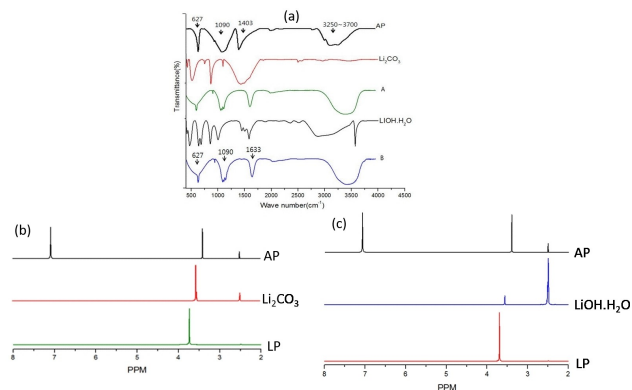


Figure 3. (a) FT-IR spectra of starting materials and LP obtained from Li_2CO_3 (A) and $\text{LiOH}\cdot\text{H}_2\text{O}$ (B), ^1H -NMR spectra of LP (b) synthesis from AP + Li_2CO_3 , (c) synthesis from AP + $\text{LiOH}\cdot\text{H}_2\text{O}$.

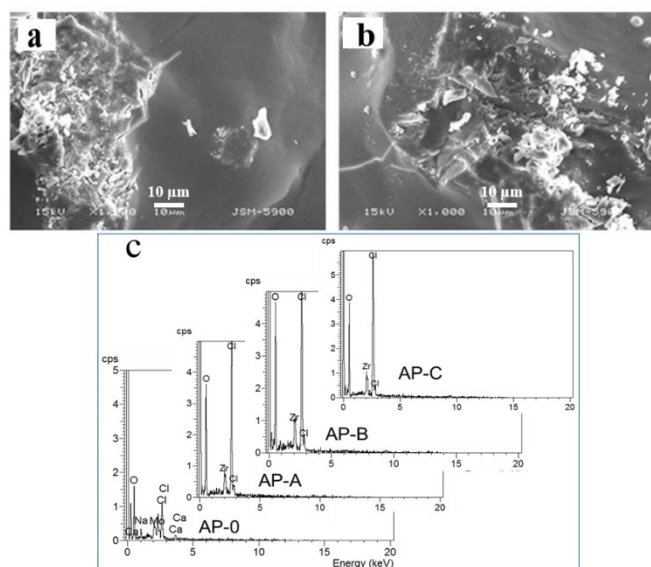


Figure 4. SEM images of (a) AP-A, (b) AP-C, and EDX spectra of purified AP.

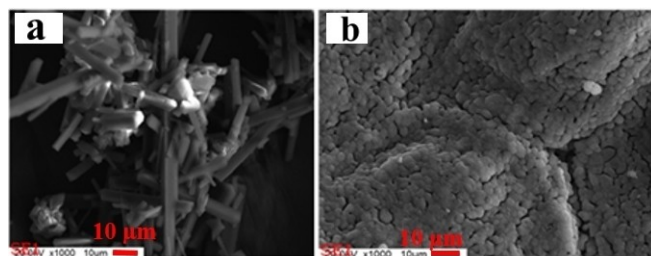


Figure 5. SEM images of LP from Li_2CO_3 (a) and $\text{LiOH}\cdot\text{H}_2\text{O}$ (b).

ple did not significantly change the level of purity. After obtaining these results, the purified material was used as a precursor for the synthesis of LP, and ICP-MS results in-

dicated a purity of synthesized LP was better purity (~99.9%), which is acceptable usage in industrial applications.

Figure 6a shows the X-ray diffraction pattern of purified AP. The diffraction peaks at angles of $2\theta = 15^\circ, 19^\circ, 21^\circ, 22^\circ, 23^\circ, 24^\circ, 25^\circ, 27^\circ$, and 30° correspond to planes (101), (011), (201), (002), (210), (102), (211), and (112), respectively. These results overlap well with JCPDS Card No. 43-0648 [30] and the tetrahedral crystal structure of AP described by Jung *et al.* [31]. All the purified AP-A, AP-B, and AP-C samples displayed better crystallinity and purity compared to crude AP. After synthesis of LP from purified AP, the XRD pattern showed (Figure 6b–c) better crystallinity with XRD characteristic peaks at $21^\circ, 23^\circ, 31^\circ, 35^\circ$, and 39° corresponding to the tetrahedral crystal structure of LP described by Lim *et al.* [32]. Further, we calculated average crystallite particle sizes of purified AP and synthesised LP samples by using Scherrer formula (Equation 1) from its corresponding XRD patterns. The results reveal the average size of crystallite particles was about more than 100 nm.

$$D = \frac{k\lambda}{\beta \cos \Theta} \quad (1)$$

Where D is crystalline domain size, λ is the wavelength of X-rays, particular peak position is θ and corresponding peak width is β [33]

4 Conclusion

In summary, we successfully purified AP from crude AP extracted from propellant motors through activated carbon purification, filtration, and recrystallization. The yield of purified AP after recrystallization was 85 %. Purity, crystallinity, and chemical composition of purified AP were investigated through various analytical techniques such as NMR, FT-IR, XRD, and ICP-MS. The results indicated that recrystallized

Table 1. ICP-MS analysis of AP, obtained after different filtering methods.

Element	LD (mg/L)	Total analysis of AP-O (mg/L)	After purification (mg/L)		
			AP-A	AP-B	AP-C
Ca	0.1	742.6	2.3	4.9	14.1
Pb	0.1	628.0	n.d.	n.d.	n.d.
Na	0.1	521.0	11.7	26.4	54.6
K	0.1	113.0	29.2	48.8	63.7
Fe	0.1	23.4	n.d.	n.d.	0.8
Mg	0.1	12.2	n.d.	n.d.	0.5
Al	0.1	11.4	n.d.	1.3	n.d.
Zn	0.1	10.4	n.d.	n.d.	0.8
Cu	0.1	8.6	n.d.	n.d.	n.d.
Si	0.1	6.7	n.d.	n.d.	n.d.
Sr	0.1	6.7	n.d.	n.d.	n.d.
Ni	0.1	5.3	n.d.	n.d.	n.d.

mg/L = ppm; n.d. = not detected; LD = limit of detection.

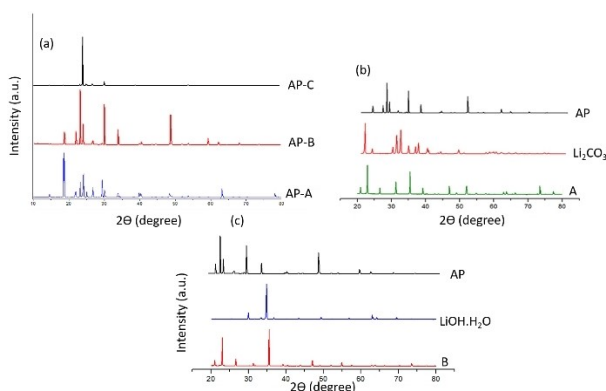
Table 2. Purity of AP obtained from different filtering methods.

Samples	AP-O	AP-A	AP-B	AP-C
Purity %	~85	99.791	99.995	99.987

Table 3. ICP-MS analysis of LP, obtained from Li_2CO_3 and $\text{LiOH}\cdot\text{H}_2\text{O}$.

Element	LD (mg/L)	From Li_2CO_3 (mg/L)	From $\text{LiOH}\cdot\text{H}_2\text{O}$ (mg/L)
Ca	0.1	n.d.	n.d.
Pb	0.1	n.d.	n.d.
Na	0.1	20	9.8
K	0.1	550	n.d.
Mg	0.1	n.d.	n.d.
Al	0.1	n.d.	n.d.
Zn	0.1	n.d.	n.d.
Li	0.1	2020	47218
Si	0.1	n.d.	49
Ni	0.1	n.d.	n.d.

mg/L = ppm; n.d. = not detected; LD = limit of detection.

**Figure 6.** XRD pattern of the (a) purified AP synthesized LP and (b) starting materials Li_2CO_3 (A) and (c) $\text{LiOH}\cdot\text{H}_2\text{O}$ (B).

AP had an excellent purity of about 99.99% compared to crude AP. Further, purified AP was reacted with Li compounds Li_2CO_3 and $\text{LiOH}\cdot\text{H}_2\text{O}$ to produce LP, which was subsequently characterized by various analytical methods. We illustrated a new strategy to transform obsolete ammunition into energy storage material, where ammonium perchlorate (used as an oxidizer) is converted into lithium perchlorate (used as an electrolyte in lithium-ion batteries). This process provides new possibilities for recycled AP and purified AP products could be effectively used as starting material for synthesis of LP.

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Data Availability Statement

No data available.

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