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Al_2O_3 Recovery From Waste Tetra Pak Packages

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Aluminum,
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Abstract: In this study, it was aimed to obtain Al_2O_3 from waste Tetra Pak packages by using the hydrometallurgical method. Tetra Pak recycling has become an increasingly researched topic around the world. Cellulose, polyethylene and aluminum, which form the structure of Tetra Pak packages, are raw materials that can be reused after recycling. Cellulose was separated from Tetra Pak's structure by hydropulping process and polyethylene was separated from Tetra Pak's structure by leaching with HCl, and optimum parameters of these processes were investigated in experimental studies. Aluminum in the structure was dissolved in acid solution with 100% efficiency in the experiments carried out with 12.076 M and 9.057 M acid concentration. Aluminum, which was dissolved in acid solution, was obtained as the $AlCl_3$ phase and precipitated as $Al(OH)_3$ with the addition of 100% stoichiometric NaOH, and the precipitates were calcined at 950 °C for 1 hour to obtain the Al_2O_3 structure. The materials used in the experimental studies and the products obtained as a result of these experiments were analyzed by Atomic Absorption Spectrometry (AAS), Fourier Transform Infrared (FTIR) Spectroscopy, Scanning Electron Microscopy (SEM) / Energy-Dispersive X-ray Spectroscopy (EDS), and optical microscopy techniques.

Atık Tetra Pak Paketlerinden Al_2O_3 Geri Kazanımı

Anahtar Kelimeler

Alüminyum,
Tetra Pak,
Geri dönüşüm

Öz: Bu çalışmada atık Tetra Pak ambalajlarından hidrometalurjik yöntem kullanılarak Al_2O_3 elde edilmesi amaçlanmıştır. Tetra Pak geri dönüşümü, dünya çapında giderek daha fazla araştırılan bir konu haline gelmiştir. Tetra Pak ambalajlarının yapısını oluşturan selüloz, polietilen ve alüminyum, geri dönüştürülerek yeniden kullanılabilen hammaddelerdir. Tetra Pak'ın yapısından selüloz hidropulping prosesi ile, polietilen ise HCl ile liç prosesiyle ayrıştırılmış ve deneysel çalışmalarda bu prosesler için optimum parametreler araştırılmıştır. Yapıdaki alüminyum, 12,076 M ve 9,057 M asit konsantrasyonuyla yapılan deneylerde %100 verimle çözünmüştür. Asit çözeltisinde çözünen alüminyum, $AlCl_3$ fazı olarak elde edilmiş ve %100 stokiyometrik NaOH ilavesiyle $Al(OH)_3$ olarak çöktürülmüştür. Bu çökeltiler Al_2O_3 yapısı elde etmek için 950 °C'de 1 saat kalsine edilmiştir. Deneysel çalışmalarda kullanılan malzemelerin ve bu deneyler sonucunda elde edilen ürünlerin analizleri Atomik Absorbsiyon Spektroskopisi (AAS), Fourier Dönüşümlü Kızılötesi (FTIR) Spektroskopisi, Taramalı Elektron Mikroskobu (SEM) / Enerji Saçılımlı X-Işını Spektroskopisi (EDS) ve optik mikroskopi teknikleriyle gerçekleştirilmiştir.

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1. Introduction

One of the biggest challenges faced by the modern world is the efficient use of organic waste consisting of plastic, rubber and solid waste [1]. Besides the recycling of waste, their volume needs to be reduced to create appropriate storage conditions [2]. The solid waste consists of organic materials, paper, cardboard, plastic, glass, aluminum and other metals [3]. One of these waste materials is Tetra Pak. It is generally called Tetra Pak based on the name of the first manufacturer, and it is widely used as aseptic packages for beverages such as milk and juice [1]. Tetra Pak packages allow perishable products to be stored for 6 months without the need for cooling [4].

Tetra Brik™ aseptic package is produced by Tetra Pak Company and used for packaging after the ultra-pasteurization process. This package consists of six layers mainly made of three materials, and these materials are cellulose (73% by weight), low-density polyethylene (LDPE, 20% by weight) and aluminum (5% by weight). Small amounts of printing ink, coatings and adhesives are also present in the structure (2% by weight) [4,5]. Each component (cellulose, polyethylene, aluminum) of the used Tetra Pak package becomes the raw material for the new product. Considering the environmental effects and limited raw material sources, it is important to recycle these materials without generating waste.

Aluminum is the most produced metallic material after iron and steel [6]. Aluminum is used in industries such as metallurgy, automotive, packaging, electric-electronics, aircraft-space, machine technologies and construction [7]. The densities of iron, copper and zinc are 7.78 g/cm^3, 8.93 g/cm^3 and 7.14 g/cm^3, respectively. On the other hand, the density of aluminum is 2.69 g/cm^3. Due to this advantageous feature, the use of aluminum in industries such as automotive, aerospace and marine industries, where weight is very important, is increasing day by day [8]. The low weight of aluminum reduces fuel consumption, which leads to low CO_2 emissions. Today, 19% of the greenhouse gas emission to the atmosphere originates from the transportation industry [9]. Aluminum's melting point is 660.25 °C, boiling point is 2467 °C, linear thermal expansion coefficient is 2.39E-07 cm/cm/°C (at 0 °C), thermal expansion is 23.1µm/(m.k) (at 25 °C), density is 2.69 g/cm^3, specific heat is 0.9 J/gK and modulus of elasticity is 76 GPA [10]. Due to these features, aluminum is used in many applications in the industry.

Aluminum can be produced as thin foil, it can be shaped easily, it prevents the passage of water, gas, steam and microorganisms, it is not affected by weather change and the environment, and it is protective against ultraviolet and infrared rays. Therefore, aluminum is used in Tetra Pak packages [1-3].

The mineral used in primary aluminum production is bauxite [11]. Alumina plants are usually built next to bauxite ores. Bauxite ore extracted from the mine is treated with sodium hydroxide melt to obtain aluminum hydroxide. The non-melting residues (red mud) formed as a result of this process are separated and alumina (aluminum oxide) is obtained by calcination of aluminum hydroxide [12]. In this context, the use of alumina is also important. Alumina is used as a raw material in the ceramic industry, glass industry, refractory materials, abrasive material production, primary aluminum production, transparent armor production, and cutting tool insert production [13]. Considering that bauxite ores in the world are limited, to meet the increasing demand for aluminum, different alternatives must be found, and aluminum used in every field must be recycled [12,14]. Tetra Pak packages containing aluminum are recycled by a process called hydropulping [15]. In this process, the separation of the cellulosic fibers takes place in a washing device using a sieve with two chambers. In the two-chamber sieve system, aluminum containing polyethylene is collected in the inner chamber, and pulp (cellulosic fibers) is collected in the outer chamber. In the hydropulping process, thin plastic and aluminum layers are kept in a waterproof membrane and separated from cellulosic fibers by centrifugal forces. The high-quality cellulosic fibers obtained are used in the production of products such as paper towels and writing paper [16]. Low-density polyethylene and aluminum residues are extruded to obtain pellets [15,17]. The utilization of these residues can be carried out by using three different methods. These methods are; i) aluminum recovery in pyrolysis furnaces, ii) recovery of polyethylene and aluminum with plasma technology, iii) processing of polyethylene and aluminum mix to obtain high-quality paper products [5,18]. Tetra Pak pyrolysis is performed in furnaces operating under inert atmosphere conditions at temperatures ranging from 350 °C to 500 °C. The products obtained as a result of pyrolysis are aluminum, gas, oil and coal [19]. Another method used to separate polyethylene and aluminum is plasma technology. In this method, plasma with a temperature of 15000 °C is sprayed to heat the polyethylene and aluminum layers together [20]. The main motivation of this study is to use a method with low energy requirement as an alternative to these methods with high energy requirements.

According to Tetra Pak Company, there is still a lack of infrastructure for the management of the domestic waste disposed of by storage. The global recycling of Tetra Pak packages used increased by 10% in 2012, from 528 tons to 581 tons. The production of recyclable materials with new production methods allows the creation of new companies and official jobs, as well as the use of recycled products. However, inadequate management of waste can pose significant risks for public health and the ecosystem [3]. In this study, in line with these targets, it was aimed to determine the optimum parameters to be used in the recovery of aluminum oxide with HCl acid leach from Tetra Pak packages.

2. Material and Method

2.1. Material

Waste Tetra Pak packages were used as the aluminum source in this study. Tetra Pak packages were cleaned and contaminations were removed. The dimensions of the cleaned samples were reduced to obtain a homogeneous mixture. Then, the samples, which were reduced in size, were mixed and as a result of this process, a homogeneous structure with the characteristics of all scraps was obtained.

The chemical composition of Tetra Pak scraps, cleaned Tetra Pak scraps, and pelletized Tetra Pak packages in the literature are shown in Table 1.

Table 1. Compositions of Tetra Pak samples (weight %) [3]

Material	Cellulose	Polythene	Aluminum
Tetra Pak (Scrap)	75	20	5
Tetra Pak (Cleaned)	0	83	17
Tetra Pak (Pellet)	0	95	5

Sigma-Aldrich HCl acid was used to separate the components of the samples. Technical grade NaOH was used to precipitate $AlCl_3$, which passed into the acid solution, and aluminum was precipitated in the hydroxide form. Precipitated aluminum hydroxide was calcined to obtain Al_2O_3.

2.2. Method

In the first stage of the experimental studies, PE-Al and cellulose, which are the components of Tetra Pak packages, were separated from each other by the hydropulping process according to the flow chart given in Figure 1. The hydropulping method used by Akoglu et al. was optimized and used [21]. Initially, Tetra Pak packages were cut into squares with an average edge length of 2.5 centimeters, then the mixture was washed with water at room temperature for 15 minutes to remove food residues from the packages. Arcelik K-8140 TB blender was used to perform the grinding process. Then PE-Al and cellulose phases were subjected to wet screening consisting of three steps. In screening and wet screening processes, PE-Al and cardboard phases were separated from each other. The screen used in the first screening step was 4 mm in size and its screen opening was 0.125 mm. In the second screening step, the screen used was 2 mm in size and its screen opening was 0.125 mm.

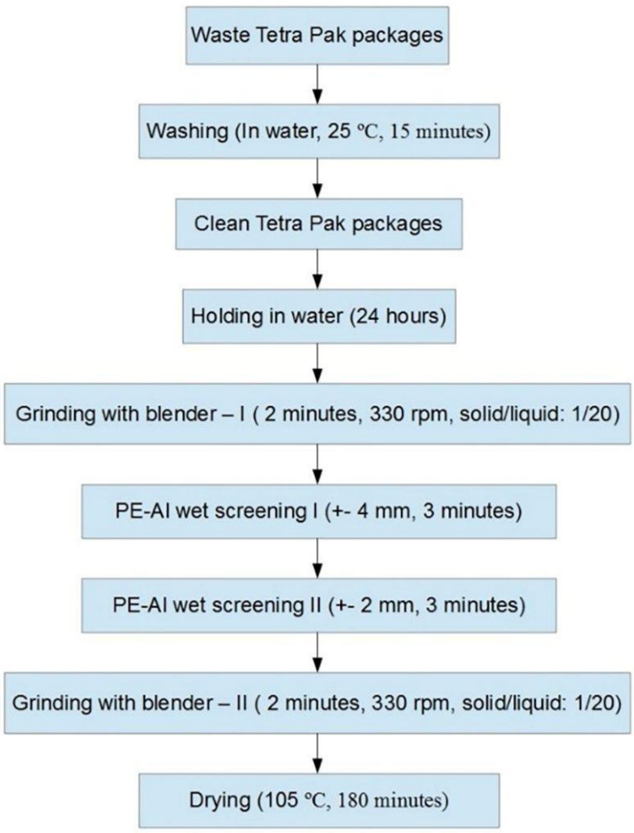

Figure 1. Hydropulping process flow chart

After the cellulose and PE-Al were separated from each other by the hydropulping method, a second process was applied to separate the polyethylene and aluminum. In the second stage of the experimental studies, the optimum parameters for dissolving aluminum in acid solution by treating the obtained PE-Al phase with HCl acid (wt. 37%) were investigated. The experiment sets executed in the second stage were divided into two. In the first set of experiments, the effects of acid molarities on the leaching efficiency were investigated (Table 2).

Table 2. Parameters applied in the experiment set investigating the effects of acid molarities in PE-Al separation

Experiment	HCl, ml	H_2O, ml	Molarity, M
1	15	45	3.019
2	30	30	6.038
3	45	15	9.057
4	60	0	12.076

In the experimental set in question, fixed parameters for 1 g of PE-Al (wt. 18.94% Al), which was used as solid phase, were; 25 °C test temperature (room temperature), 20 minutes leaching time, 450 rpm stirring rate and 1/60 solid to liquid ratio.

In the second set of experiments, in experimental studies carried out at low acid molarities (3.019 M and 6.038 M), the effects of temperature on the leaching efficiency were investigated (Table 3). Experiments were carried out at 35 °C, 45 °C and 55 °C, respectively, based on the HCl acid concentrations used in experiments 1 and 2 (3.019 M and 6.038 M). Fixed parameters in the second set of experiments were 20 minutes leaching time and 450 rpm mixing speed.

In the experimental studies, Equation 1 was used to calculate the leaching efficiency (LE).

$$LE = ((Mass\ of\ aluminum\ passed\ into\ acid\ solution, g)\ /\ (Aluminum\ quantity\ in\ PE - Al, g))\ x\ 100 \quad (1)$$

Table 3. Experimental parameters used in investigating the effect of temperature on the leaching efficiency in PE-Al separation

Experiment	HCl, ml	H_2O, ml	Molarity, M	T, °C
5	15	45	3.019	35
6	15	45	3.019	45
7	15	45	3.019	55
8	30	30	6.038	35
9	30	30	6.038	45
10	30	30	6.038	55

2.3. Analysis methods

Fourier Transform Infrared (FTIR) Spectroscopy (Perkin Elmer Spectrum 100) was used to understand the structure of precipitates. FEI Inspect S50 Scanning Electron Microscope (SEM) and EDAX Octane Prime Energy-Dispersive X-ray Spectroscopy (EDS) were used to examine the crystallographic structures of the samples. Optical microscope micrographs of the prepared samples were taken with the LEICA Optical Microscope before the hydropulping process. Chemical analyses were performed with Perkin Elmer AAnalyst 800 Atomic Absorption Spectrometer (AAS).

3. Results and Discussion

In the first stage of the experimental studies to obtain aluminum from waste Tetra Pak packages, cellulose / PE-Al mix was separated from each other by hydropulping process carried out in accordance with the flow chart shared in Figure 1. Before this process, optical microscope micrographs were taken to observe that if samples were completely free from contaminants. Optical microscope micrographs are given in Figure 2. As seen in Figure 2, there were no contaminants on the inner and outer surfaces of the samples, and there was metallic aluminum on the inner surface. The PE-Al residues obtained after the hydropulping process are shown in Figure 3. It is seen that the samples were completely free from cellulose.

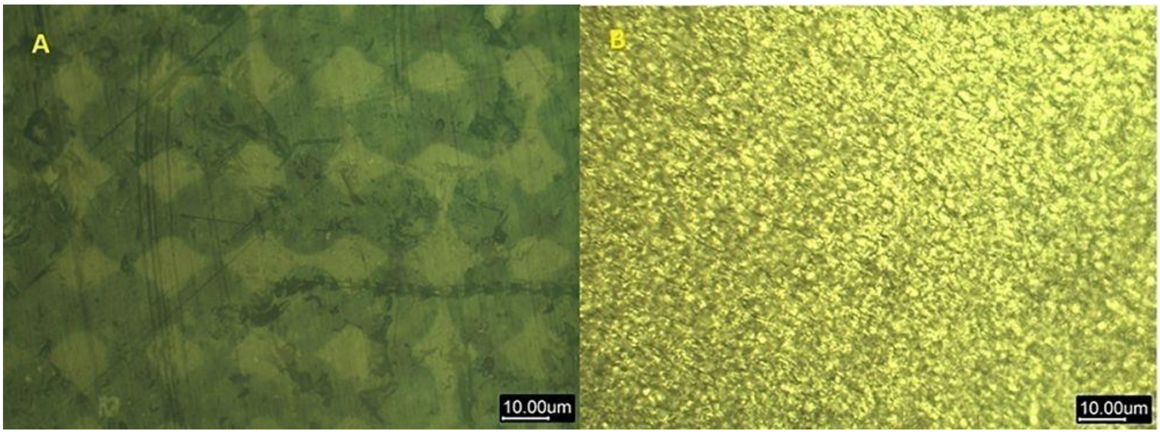

Figure 2. Optical microscope micrographs of the Tetra Pak package; a) the outer layer of the Tetra Pak package (layer with the pattern on it), b) the inner layer of the Tetra Pak package

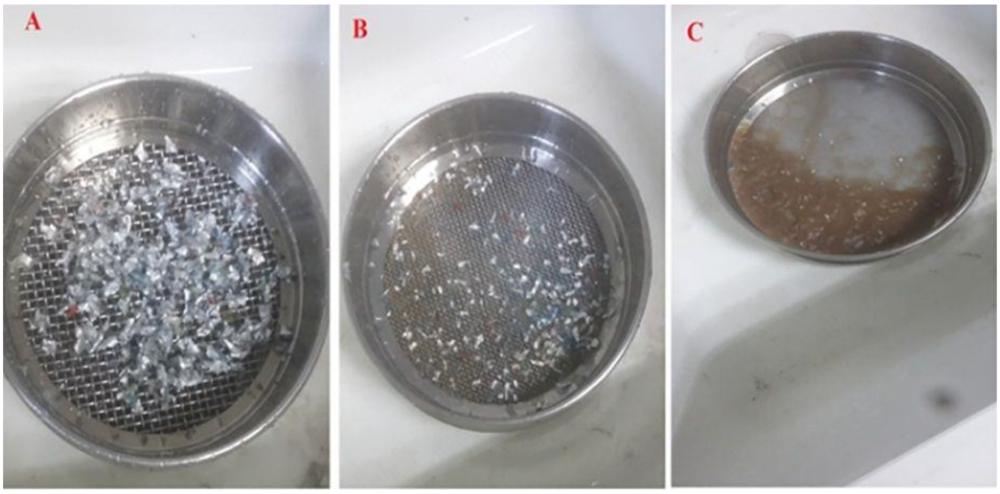

Figure 3. End products obtained as a result of hydropulping process; a) PE-Al phase, b) cellulose pulp and PE-Al phase, c) cellulose pulp

The content of the PE-Al phase was measured by the chemical analysis method. As a result of the analysis, it was determined that the chemical content of the material was 79.92% polyethylene and 20.08% aluminum by weight. The average width of the PE-Al lamels was found to be 1.9 mm.

In the second stage, the process of separating Al from the PE-Al layer, which was separated from cellulose in the first stage, was carried out hydrometallurgically. Experiments of dissolving aluminum in acid solution were executed at room temperature for the increasing acid molarities, and increasing temperatures for low acid molarities. When the experiment results were examined, it was determined that the amount of aluminum dissolved in acid solution increased with increasing acid molarity. The results are seen in Figure 4 and Table 4. When Figure 4 is examined, it is seen that the leaching efficiency was over 100% at 9.057 M and 12.076 M acid concentrations. This is due to the probability of relatively low pulse and low homogeneity of the sample which was weighed as 1 g. This situation is defined as the ingot effect in metallurgy and is frequently encountered in the analysis of Au-Ag ores. Therefore, experiments at 9.057 M and 12.076 M acid concentrations, where 100% efficiency was exceeded, provided sufficient conditions. Sufficient conditions were those where 100% efficiency was achieved.

Table 4. The effects of HCl acid molarity and temperature on the leaching efficiency of aluminum

Experiment	Molarity	Temperature, °C	Al leaching efficiency, %
1	3.019	25	7.40
2	6.038	25	61.25
3	9.057	25	100.00 (116.68)
4	12.076	25	100 (100.42)
5	3.019	35	6.07
6	3.019	45	14.78
7	3.019	55	15.42
8	6.038	35	54.96
9	6.038	45	63.41

10	6.038	55	89.76

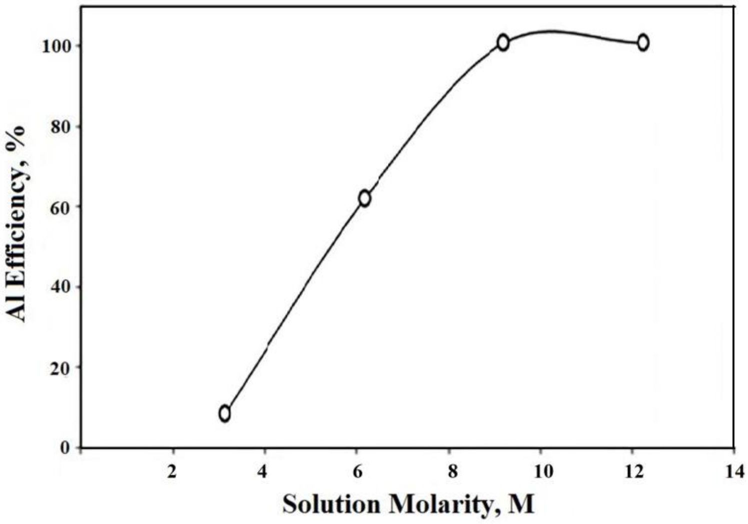

Figure 4. The effect of HCl acid molarity on the leaching efficiency of aluminum

To increase the leaching efficiency of aluminum at 3.019 M and 6.038 M acid concentrations, experiments were repeated at higher temperatures and the same molarity values, and the results were shared in Figure 5. It was observed that the leaching efficiency of aluminum increased with increasing experiment temperatures. The highest efficiency in this set of experiments was obtained as 89.76% at 55 °C, which was the highest temperature used in the experiments. In experiments carried out at an acid concentration of 3.019 M, it was determined that the leaching efficiency of aluminum did not exceed 20% despite the increase in temperature. According to these data, it was determined that the acid molarity of the solution must be 6.038 M as a minimum.

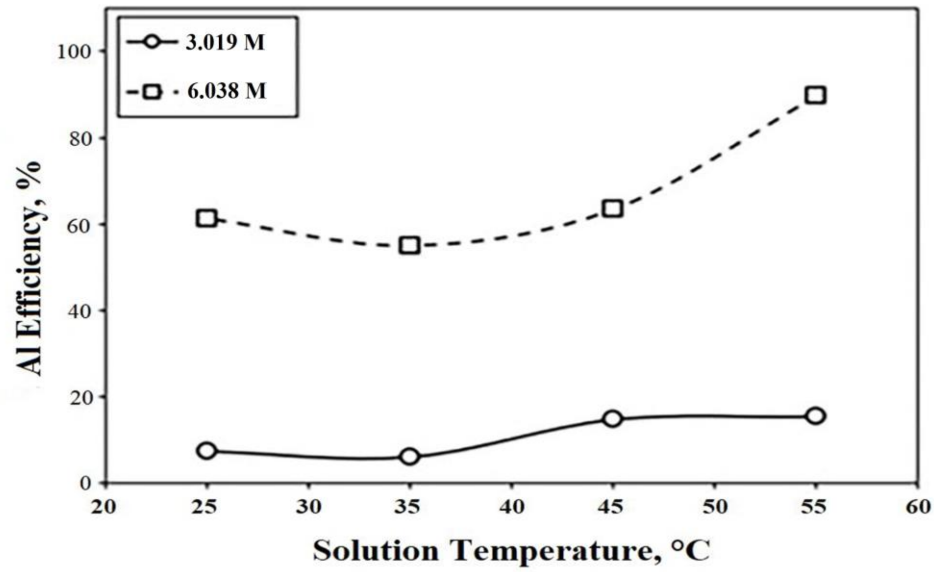

Figure 5. The effect of temperature on the leaching efficiency of aluminum

In the studies conducted, Atomic Absorption Spectrometer (AAS) was used to detect the remaining Al in PE-Al samples which were treated with HCl. According to the analyses results, there was no aluminum in the composition of the cakes when the molar concentration of HCl acid was high, but there was a small amount of aluminum left in the system when the molar concentration of HCl acid was low (3.019 M HCl and 6.038 M HCl).

It was seen that aluminum reacting stoichiometrically with HCl, which was used in high concentrations, could easily transform into $AlCl_3$ as seen in Equation 2, then $AlCl_3$ dissolved and passed into liquid phase, and for that reason, it was not present in the cake phase.

$$2Al + 6\ HCl = 2AlCl_3 + 3H_2 \quad (2)$$

According to the reaction mechanism, excess material of the reaction between high concentration HCl and Al was HCl. Therefore, it was thought that all of the aluminum was used during the reaction and formed $AlCl_3$, and $AlCl_3$ passed to the raffinate phase with the remaining HCl. For this reason, there was no aluminum in leach cake in leaching experiments performed at high concentrations of HCl.

However, when the molarity of the acid was low (3.019 M HCl and 6.038 M HCl), it was thought that the amount of HCl reacting with aluminum was not enough for the reaction to occur 100% stoichiometrically, and therefore aluminum was not completely used. Thus, aluminum was found in the filter cake obtained after leaching. In addition, due to the large number of water molecules resulting from low concentration of HCl, formed $AlCl_3$ could react with excess H_2O in the medium in accordance with Equation 3.

$$AlCl_3 + 3H_2O = Al(OH)_3 + 3HCl \quad (3)$$

Due to the precipitated aluminum hydroxide as a result of the reaction occurred, the aluminum phase was determined in the leach cake in the process performed at a low concentration of HCl solution. Also, if there is excess water molecule in the media, it forms aluminum oxide salt with the following reaction mechanism (Equation 4).

$$2AlCl_3 + 3H_2O = Al_2O_3 + 6HCl \quad (4)$$

The aluminum oxide salt formed as a result of this reaction also precipitates, and this causes the leach cake to contain aluminum. Leaching operations were carried out at high temperatures to remove aluminum, which was seen at low molarity values, but aluminum found in leach cake could not be dissolved in acid solution. The reason for this is the low solubility constants of $Al(OH)_3$ and Al_2O_3, which are compounds predicted to occur by the reaction mechanisms described above, therefore by increasing the temperature, the solubility does not reach the level to pass the raffinate phase.

The structure of the polyethylene separated from aluminum by the hydrometallurgical process was characterized by the FTIR method and the results are given in Figure 6.

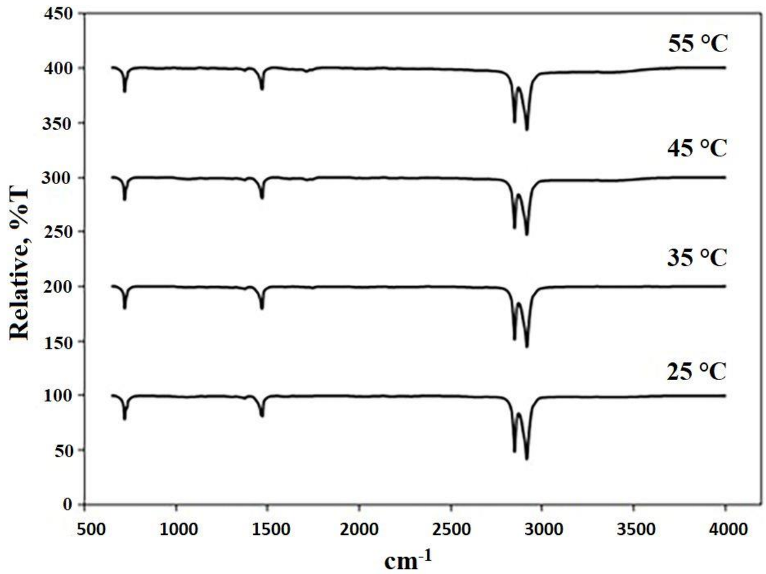

Figure 6. FTIR patterns of filter cakes obtained from experimental studies carried out with 6.038 M HCl and increasing temperatures

According to Figure 6, no impurities based on Al or other structures were found in the structure of the polyethylene obtained as a result of leaching with 6.038 molar HCl solution. Accordingly, for the solution having 6.038 M acid concentration, it was determined that at every temperature used in the experiments, aluminum was dissolved in acid solution at a high rate and completely separated from polyethylene. After determining the leaching efficiencies of aluminum, the solution with the best efficiency (25 °C, 9.057 M HCl acid concentration) was prepared and PE-Al was dissolved in acid solution under optimum conditions. 100% stoichiometric ratio of NaOH was added to the aluminum which was dissolved in acid solution and $AlCl_3$ was precipitated in $Al(OH)_3$ form. In this process, pH should be adjusted to 7. Whether the solution is acidic or basic prevents precipitation, since Al tends to remain in solution in both cases. FTIR analysis result of $Al(OH)_3$ obtained as the result of precipitation is shown in Figure 7. It is seen in the figure that $Al(OH)_3$ was successfully precipitated. However, it was determined that there was some amount of precipitated $Na[Al(OH)_4]$ in the structure because of NaOH.

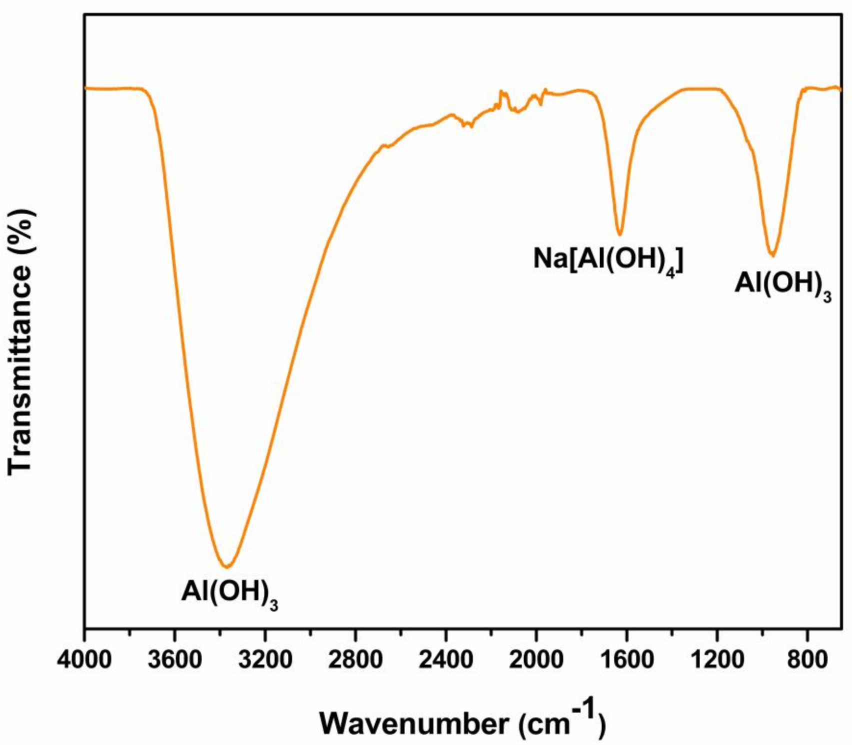

Figure 7. FTIR pattern obtained by adding NaOH to filter cakes in experimental studies carried out with 9.057 M HCl and 25 °C of temperature

After the precipitation process, the calcination process was carried out to obtain Al_2O_3 from $Al(OH)_3$. In the calcination process, samples were calcined at 950 °C for 1 hour, and the FTIR pattern of the obtained products is shown in Figure 8. When Figure 8 is examined, it was determined that Al_2O_3 was successfully obtained and some amount of aluminum hydroxide remained in the structure.

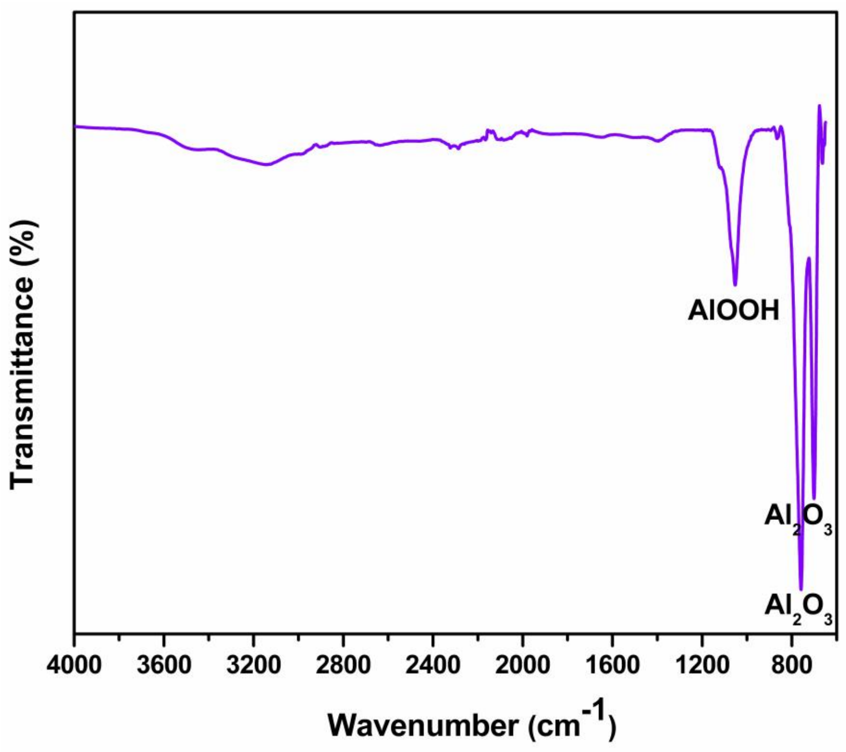

Figure 8. FTIR pattern of calcined Al_2O_3

SEM analyses were performed to examine the microstructures of precipitated and calcined samples. The SEM image of $Al(OH)_3$ is seen in Figure 9, and the EDS analysis is given in Table 5. The SEM image of Al_2O_3 obtained as a result of calcination is seen in Figure 10, and the EDS analysis is shared in Table 6. EDS analysis shared in Table 5 shows that a low amount of sodium remained in the structure in parallel with the FTIR results. According to the results of EDS analysis shared in Table 6, as a result of the calcination process; Na could not be eliminated, the amount of H, which was 36.60%, decreased to 3.37% and the amount of O_2 increased from 21.04% to 42.95%. According to these data, it was determined that Al_2O_3 was obtained successfully.

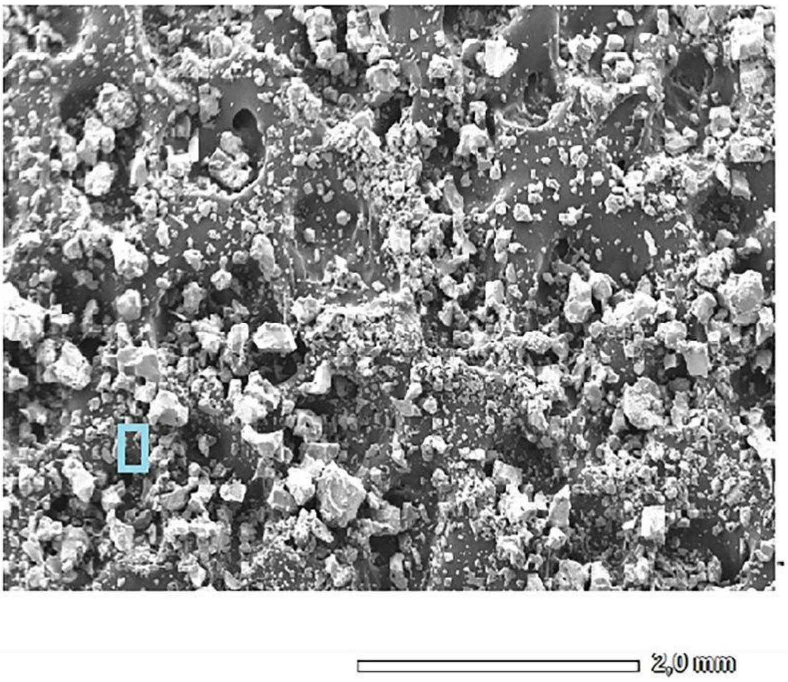

Figure 9. SEM image of $Al(OH)_3$

Table 5. EDS analysis of $Al(OH)_3$

Element	keV	Mass %	Sigma	Atom %
Al	1.486	37.56	0.59	51.47
O	0.525	21.04	0.27	20.06
Na	1.041	4.80	0.23	12.02
H	2.621	36.60	0.24	16.45
Total		100.00		100.00

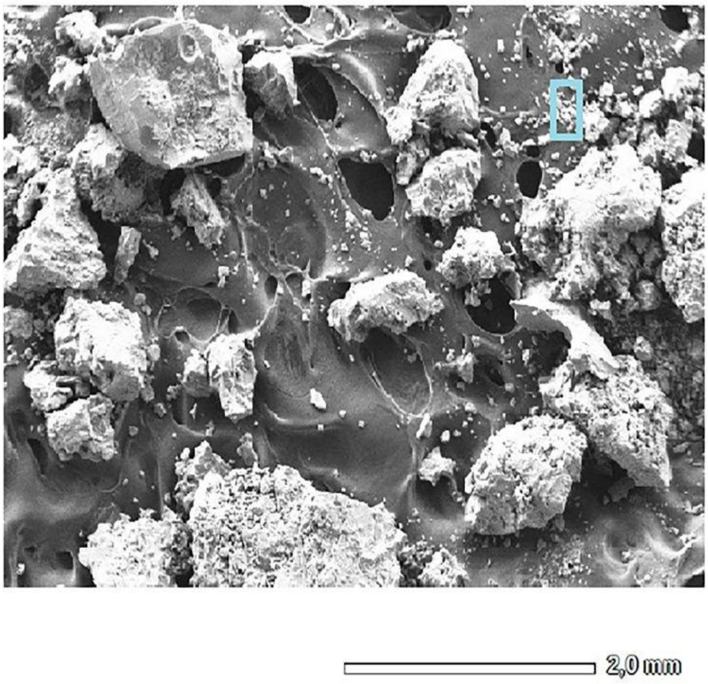

Figure 10. SEM image of calcined Al_2O_3

Table 6. EDS analysis of calcined Al_2O_3

Element	keV	Mass %	Sigma	Atom %
Al	1.486	52.65	0.40	55.94
O	0.525	42.95	0.19	42.47
Na	1.041	1.03	0.17	0.21
H	2.621	3.37	0.18	1.38
Total		100.00		100.00

Figure 11 shows the SEM images of $Al(OH)_3$ and Al_2O_3 at different magnifications. It is seen in the figure that the hydroxide structure turns into a finer oxide structure as a result of the transformation from $Al(OH)_3$ to Al_2O_3.

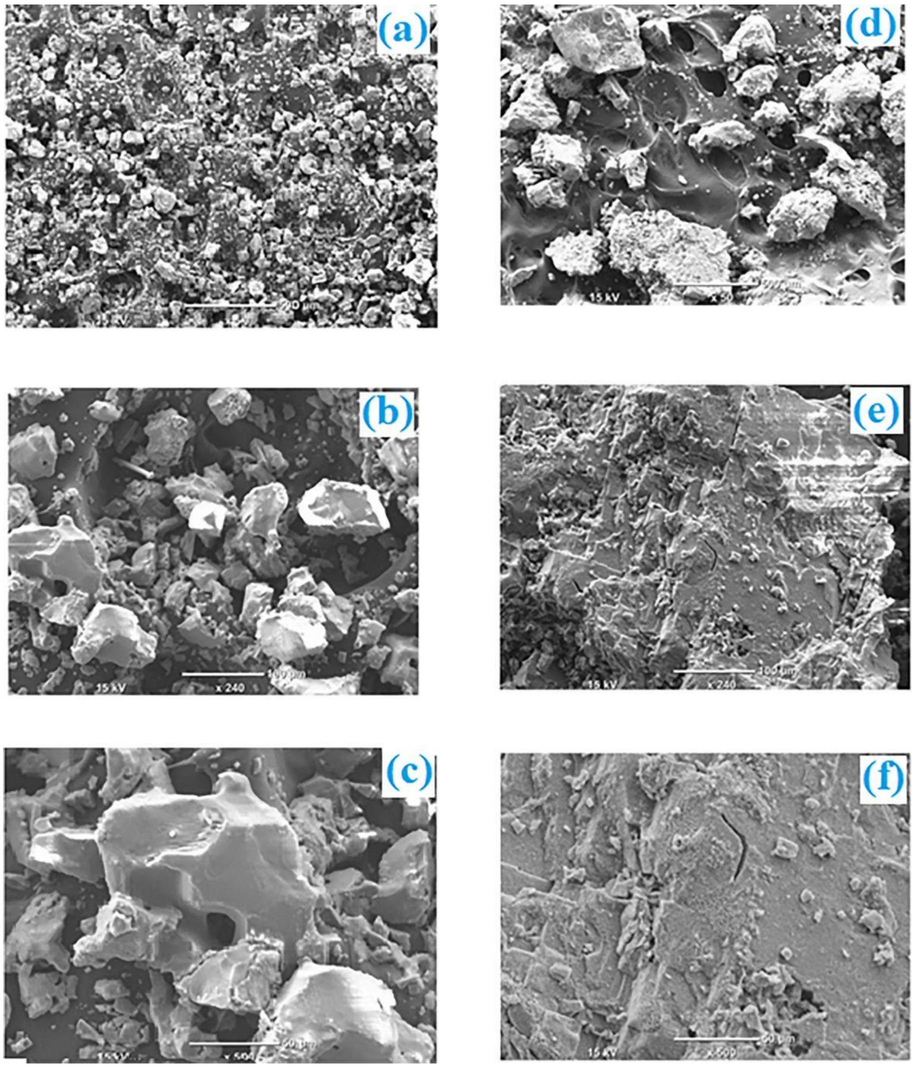

Figure 11. SEM images of $Al(OH)_3$ and Al_2O_3 at different magnifications; a) $Al(OH)_3$ at 50X magnification, b) $Al(OH)_3$ at 240X magnification, c) $Al(OH)_3$ at 500X magnification, d) Al_2O_3 at 50X magnification, e) Al_2O_3 at 240X magnification, f) Al_2O_3 at 500X magnification

4. Conclusion

In this study, optimum parameters for obtaining Al_2O_3 from waste Tetra Pak packages were determined by carrying out experimental studies.

- Waste Tetra Pak samples were purified from cellulose and contaminants by subjecting to the hydropulping process, then HCl acid leaching was applied, and aluminum was separated from polyethylene by dissolving in acid solution.
- In the experiment carried out with the parameters of 12.076 M acid concentration, 25 °C temperature and 400 rpm stirring rate, aluminum was completely dissolved in acid solution, and was obtained as the $AlCl_3$ phase.
- Then, $Al(OH)_3$ precipitation was acquired with stoichiometrically 100% NaOH addition. The $Al(OH)_3$ precipitate was calcined at 950 °C for 1 hour, and the Al_2O_3 structure was successfully obtained.

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