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Article

Obtaining and characterization of terephthalic acid via acid and basic hydrolysis of recycled poly (ethylene terephthalate)

Obtención y caracterización de ácido tereftálico mediante hidrólisis ácida y básica de poli (etilen tereftalato) reciclado

De la Cruz, Boris*[a], Domínguez, Ricardo[b] and Moreno, Juan[c]

[a] Universidad Salvadoreña Alberto Masferrer • NNH-0587-2025 • 0000-0001-7109-0793
[b] Universidad Salvadoreña Alberto Masferrer • NNH-0572-2025 • 0009-0004-3326-0148
[c] Universidad Salvadoreña Alberto Masferrer • NNH-0663-2025 • 0000-0002-2621-5267

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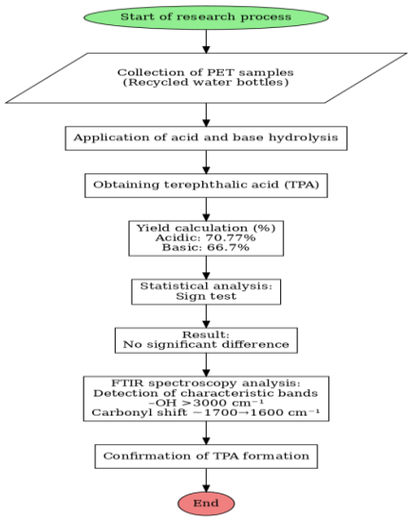
* ✉ [boris.delacruz05@liveusam.edu.sv]

Abstract

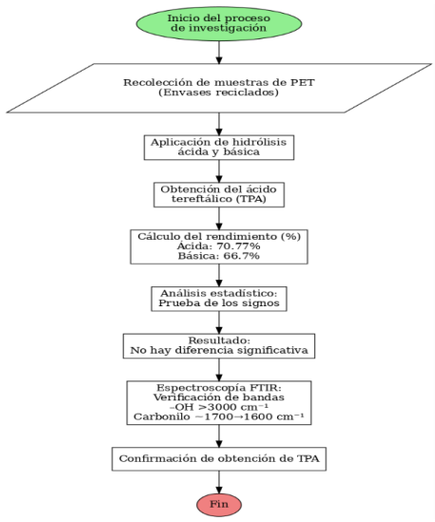
Acid-base hydrolysis is a methodology that can be used for the chemical treatment of plastic waste. This research aims to apply the mentioned methodology in poly (ethylene terephthalate) PET from reused plastic bottles to obtain terephthalic acid (TPA) under acid and basic hydrolysis conditions. Subsequently, the performance of both methodologies was compared. On average, performance percentages of 70.77% and 66.7% respectively were obtained. These data were statistically analyzed using the sign test, finding that there are no significant differences between the medians of the data sets studied. The analysis by FTIR spectroscopy of the products obtained, specifically through the appearance of the characteristic band for –OH corresponding to carboxylic acid above 3000 cm-1 and the shift of the carbonyl group band from approximately 1700 cm-1 (type ester) up to approximately 1600 cm-1 (acid) allowed us to verify the obtaining of TPA.



Hydrolysis, terephthalic acid, FTIR spectroscopy

Resumen

La hidrólisis ácido-base es un método de reciclaje utilizado para tratar residuos plásticos. Esta investigación se centró en aplicar dicha metodología en plásticos de poli (etilen tereftalato) (PET) recolectados de envases para el comercio de agua purificada, con el objetivo de obtener ácido tereftálico (TPA) bajo condiciones de hidrólisis ácida y básica. Posteriormente, se comparó el rendimiento de ambas metodologías. En promedio, se obtuvieron porcentajes de rendimiento de 70.77% y 66.7% respectivamente, estos datos se analizaron estadísticamente mediante la prueba de los signos, encontrándose que no hay diferencias significativas entre las medianas de los conjuntos de datos. El análisis por espectroscopía de FTIR, de los productos obtenidos, específicamente mediante la aparición de la banda característica para –OH correspondiente a ácido carboxílico sobre los 3000 cm^{-1} y el desplazamiento de la banda del grupo carbonilo desde aproximadamente 1700 cm^{-1} (tipo éster) hasta aproximadamente 1600 cm^{-1} (ácido) permitieron comprobar la obtención del TPA.



Hidrolisis, ácido tereftálico, espectroscopia FTIR

Area: Promotion of frontier research and basic science in all fields of knowledge

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Introduction

Plastics have revolutionized our society, enabling the cost-effective manufacturing of a variety of materials with countless applications across diverse industries. The industrial-scale production of plastics significantly began to expand around 1950, when global plastic production reached 2 million tons per year (Green Chem., 2022, 24, 8899).

Currently, the accumulation of plastics in soil and aquatic bodies constitutes a global contamination problem, driven by the excessive amount of plastic waste resulting from various anthropogenic activities in these environments. This accumulation poses a serious risk to the environment and ecosystems, as plastics can enter the trophic chain, leading to detrimental effects such as ingestion and subsequent mortality in organisms. (Green Chem., 2022, 24, 8899; Castaño et al., 2018).

As of 2015, an astonishing 8.3 billion metric tons of plastics were estimated to have been manufactured. Furthermore, in 2017, 35.4 million tons of plastic were produced in the United States. To date, billions of tons of plastics have been produced, and the progressive accumulation of plastic waste over the past decades has contributed significantly to this issue (Lucas et al., 2021; Tessa et al., 2020; Kayee et al., 2021).

Poly(ethylene terephthalate) (PET), polyethylene (PE), polypropylene (PP), polystyrene (PS), polyvinyl chloride (PVC), and nylon (NYL) are the most common polymers present in plastic waste, primarily in both terrestrial environments and marine life (Diaz-Silvarrey et al., 2018; Dorin & Danuta, 2021). Their massive use has led to a significant increase in plastic waste, with an average annual growth rate of 8.7% (Mecozzi & Nisini, 2019). Given the depletion of oil resources and the increasing environmental impact, the recovery of monomers from plastic waste has become more necessary than ever. (Diaz-Silvarrey et al., 2018; Mecozzi & Nisini, 2019).

El Salvador, like many other nations globally, exhibits significant consumption of plastic material, the fragmented particles of which accumulate in various ecosystems, including the littoral zone.

Specifically, the occurrence of microplastics in Pacific Ocean surface water was confirmed within the Los Cóbanos Protected Natural Area in El Salvador. Furthermore, in 2017, a total of 21.7 g of plastic waste was reported within a 38,400 cm^2 area across different beaches along the country's coast, based on measurements conducted during the 2016 wet and dry seasons. (Barraza, 2017).

"In the European Union (EU) and the United Kingdom (UK), plastic waste is estimated to account for up to 10–13% of Municipal Solid Waste (MSW), of which 7% (1.7 million tons) is Poly(ethylene terephthalate) (PET). Furthermore, of the plastic waste generated in the United States, 5 million tons was composed of PET (Tessa et al., 2020; Diaz-Silvarrey et al., 2018).

PET is a linear thermoplastic polymer formed by the reaction of terephthalic acid (TPA) and ethylene glycol (EG), or through the transesterification of dimethyl terephthalate and EG (Alnaqbi et al., 2015), PET is widely utilized in textiles, polyester fibers, food packaging, plastic films, electronic devices, mechanical equipment, and other fields, owing to its favorable properties such as chemical and thermal resistance, low CO2 permeability, and lightness, among others (Yang et al., 2021; Barredo et al., 2023; Raheem et al., 2019; Čolnik et al., 2022; RSC Adv., 2018, 8, 8209). The chemical structure of the PET monomer unit is shown in Figure 1:

Box 1

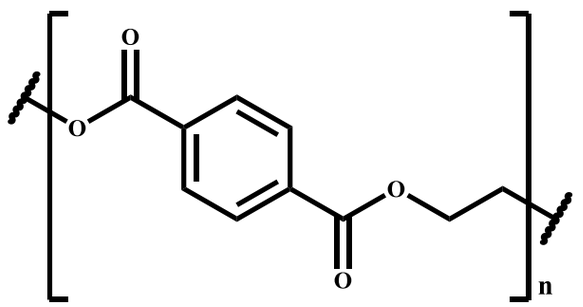

Figure 1
Chemical Structure of Polyethylene Terephthalate
Source: Authors

The majority of global PET manufacturing is allocated to the production of synthetic fibers (over 60%), while bottle production accounts for approximately 30% of the worldwide demand (Cosimbescu et al., 2021).

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The final disposal of PET is typically recycling, which is carried out in different plants that process the material for re-employment, such as in the packaging industry. However, recycled PET (rPET) cannot be used directly for food packaging due to hygiene concerns (Barraza, 2017; Ortiz-Pech et al., 2020; Das & Tiwari, 2019). In turn, various methodologies exist for the treatment of plastic waste such as PET, including mechanical, chemical, and enzymatic methods, among others. The chemical modification of PET represents an alternative for the final disposal of this material, since its monomers (TPA and EG) can serve as a starting point for obtaining materials with a diversity of applications (Dorin & Danuta, 2021; RSC Adv., 2018, 8, 8209).

One of the most promising chemical processes for the depolymerization of PET is hydrolysis. This process, whether conducted in the laboratory or in industrial plants, is typically performed at high temperatures and pressures under acid, basic, or neutral catalysis (Dorin & Danuta, 2021).

The basic hydrolysis of PET (Figure 2) is typically carried out using an aqueous solution of NaOH or KOH at a concentration of 4–20 wt% (Al-Sabagh et al., 2016). The reaction products are EG and the disodium or dipotassium terephthalate salt (TPA-Na_2 and TPA-K_2, respectively). Pure TPA can then be obtained by neutralizing the reaction mixture with a strong mineral acid (for example, H_2SO_4) (Karayannidis et al., 2002).

Box 2

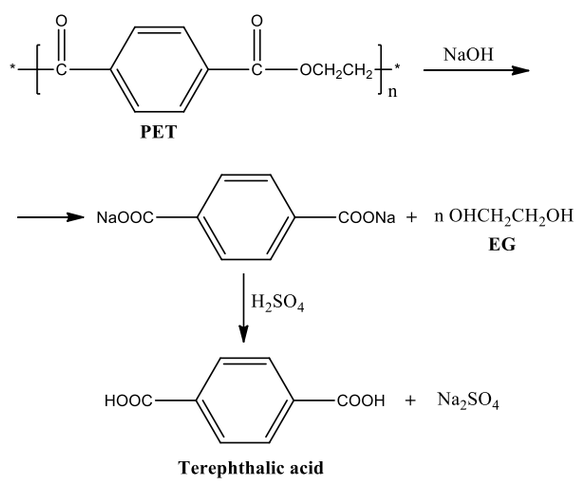

Figure 2
Basic Hydrolysis of PET using NaOH or KOH, adapted from [20]
Source: Authors

In the acid hydrolysis of PET waste, H_2SO_4, HNO_3, HCl, and H_3PO_4 are commonly used (Al-Sabagh et al., 2016; Islam et al., 2023). Acid hydrolysis is most frequently performed using concentrated sulfuric acid, as shown in Figure 3 (Al-Sabagh et al., 2016).

Box 3

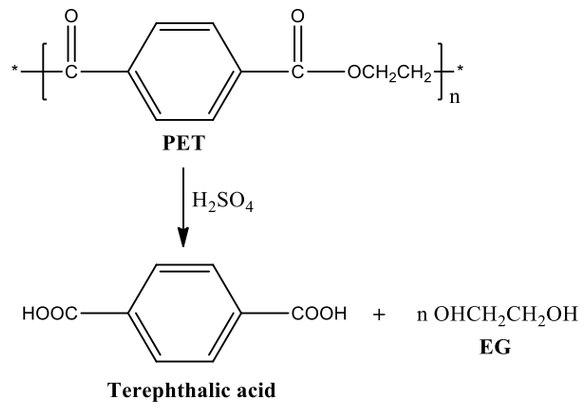

Figure 3
Basic Hydrolysis of PET using H_2SO_4 adapted from [20]
Source: Authors

The reaction products are EG and TPA. However, the process proves costly due to the requirement for recycling large quantities of concentrated H_2SO_4 and the subsequent purification of EG from the sulfuric acid (Al-Sabagh et al., 2016).

The purpose of this research is to perform the chemical recycling of PET bottles on the campus of the Universidad Salvadoreña Alberto Masferrer, by comparing straightforward basic and acid hydrolysis methodologies and employing a statistical test to determine which hydrolysis type yields the highest conversion percentage of PET to TPA. The ultimate goal is to present an alternative option for the recycling and treatment of PET-based plastic waste in El Salvador.

Methodology

Materiales

During the experimental development of this research, the following reagents were used: Sodium hydroxide pellets (P.A., Merck, Lot 106498, CAS 1310-73-2), sulfuric acid 95–98 wt% (P.A., Merck, Lot 100731, CAS 7664-93-9), butan-1-ol (P.A., Merck, Lot 101990, CAS 71-76-3), and potassium hydroxide flakes (P.A., Merck, Lot 105029, CAS 1310-58-3).

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PET Samples

PET derived from fragmented plastic bottles collected at the Universidad Salvadoreña Alberto Masferrer was used. The inclusion criterion for the plastic bottles required them to be colorless and composed exclusively of PET. These bottles were subsequently washed with soap and water and cut into fragments of approximately 0.25 cm^2.

Basic Hydrolysis of PET

Based on the methodology described by (Ramírez et al., 2010), the basic hydrolysis of PET was carried out by mixing approximately 1 g of PET with 40 mL of the 3.34 M KOH/1-butanol solution, as illustrated in Figure 4.

Box 4

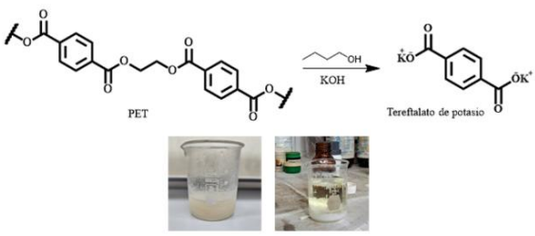

Figure 4
Basic Hydrolysis Process of PET

Source: Authors

The previously prepared mixture was maintained under constant stirring and heating at 110 °C for 50 minutes. Following this period, a white solid was obtained within the reaction mixture. To the previously described mixture, 40 mL of distilled water was added to dissolve the formed solid. From this new mixture, the aqueous phase was extracted and acidified with concentrated H_2SO_4 (Figure 5), resulting in the precipitation of the product. The product was subsequently isolated by filtration, washed with abundant distilled water, and analyzed using FTIR.

Box 5

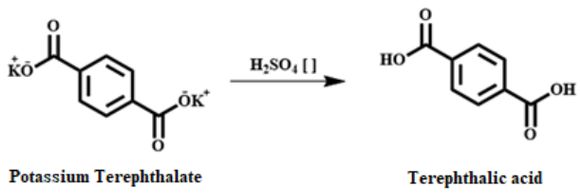

Figure 5
Neutralization equation of the dipotassium salt of terephthalic acid

Source: Authors

Acid Hydrolysis of PET

Following the methodology described by (Morales Palomino, 2010), acid hydrolysis of PET was carried out, a process shown in Figure 6. For this, approximately 1 g of PET was mixed with 40 mL of concentrated H_2SO_4. The resulting mixture was heated within a temperature range of 90-100 °C for 40 minutes, after which the complete dissolution of the plastic was observed. Subsequently, the mixture was cooled to room temperature and washed multiple times with distilled water until neutral pH was reached and no further precipitation of a white solid was observed. The obtained solid was filtered, washed with abundant distilled water, and finally analyzed using FTIR.

Box 6

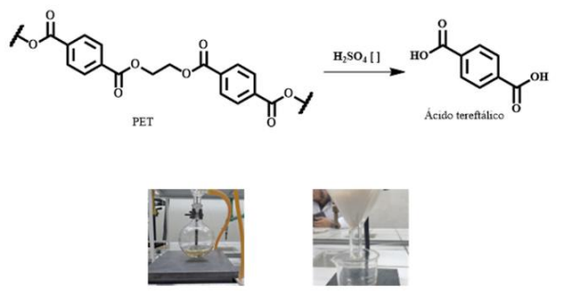

Figure 6
Acid hydrolysis process of PET

Source: Authors

FTIR Characterization

The Fourier-Transform Infrared (FT-IR) spectra of the terephthalic acid obtained from PET hydrolysis were recorded using a Thermo Scientific Nicolet iS5 equipped with an Attenuated Total Reflectance (ATR) unit (iD7), covering the range of 500 to 4000 cm^{-1}.

Statistical Analysis

Statistical analysis was performed using R (version 4.3.3, released in February 2024), an open-source software under the GNU General Public License. This environment was chosen due to its cost-effectiveness, flexibility, and the extensive tools it offers for conducting reproducible analyses and high-quality visualizations. Given the small sample size and the nature of the data, non-parametric methods were applied, specifically the sign test, to compare the medians of the yields obtained under acid and basic hydrolysis conditions.

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This statistical approach allowed for the evaluation of significant differences without requiring strict assumptions regarding data distribution. Furthermore, descriptive plots such as boxplots were generated, which clearly visualized the variability, interquartile ranges, and differences between both experimental conditions. The use of R not only ensured the transparency of the analysis but also favors its reproducibility.

Results

Hydrolysis

Tables 1 and 2 show the results obtained from the acid and basic hydrolysis methodologies employed, recording the mass of product achieved as well as the respective yield percentages:

Box 7

Table 1
Yield obtained from acid hydrolysis

N°	Hydrolysis		
	Acid		
	PET (g)	TPA (g)	Yield (%)
1	1.0180	0.7267	71.39
2	1.0175	0.7147	70.24
3	1.0182	0.7193	70.69

Source: Authors

Box 8

Table 2
Yield obtained from basic hydrolysis

N°	Hydrolysis		
	Basic		
	PET (g)	TPA (g)	Yield (%)
1	1.0059	0.6574	65.35
2	1.0034	0.6715	66.92
3	1.0015	0.6604	65.94

Source: Authors

The percentage yield was calculated according to the following formula:

$$\%\text{yield TPA} = \frac{\text{g TPA}}{\text{g PET}} \times 100 \qquad [1]$$

Acid hydrolysis yielded better results concerning the recovery of TPA. This may be associated with the fact that TPA is obtained in the bulk of the reaction, whereas, in basic hydrolysis,

TPA is first obtained as the dipotassium salt. This requires a liquid-liquid extraction prior to the precipitation of the TPA, a procedure in which small amounts of the dipotassium salt may be lost, thus affecting the process yield.

Statistical analysis

To compare the two related samples, a non-parametric test was applied using the R software (version 4.3.3, released in February 2024): the sign test. This test relies on the direction of change between the data pairs, without considering the magnitude of the change. Each pair of yield results for the two types of hydrolysis were compared, and a simple count was made of how many times the acid hydrolysis sample had a higher value than the basic hydrolysis sample, and vice versa.

Subsequently, the sign statistic is used to determine if there is a significant difference between the two samples. The sign test relies on the null hypothesis that there is no median difference between the two groups (acid hydrolysis and basic hydrolysis), and the alternative hypothesis that a difference does exist.

Box 9

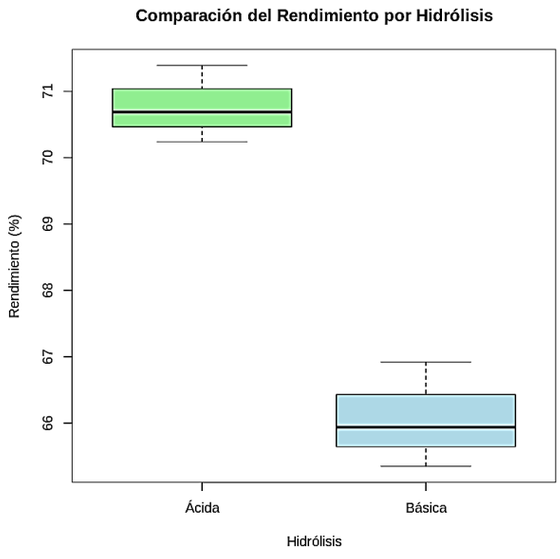

Figure 7
This graph allows a visual comparison of the medians, interquartile ranges, and possible outliers between the acid and basic hydrolysis conditions.
Source: Authors

For the yield results shown in Tables 1 and 2, three positive differences and zero negative differences were found between the acid yield and the basic yield.

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This indicates that, for all data pairs, the acid yield was consistently higher than the basic yield. Upon performing the sign test (using a binomial test, as the sign test can be considered a special case of the binomial test with p=0.5 under the null hypothesis), the resulting p-value was 0.25. This p-value represents the probability of obtaining a sign distribution as extreme as (or more extreme than) the one observed, assuming there is no true difference in the median between the two groups

Given that this p-value (0.25) is not generally considered sufficiently small to reject the null hypothesis (a threshold of 0.05 or 0.01 is typically used), there is insufficient evidence to conclude that there is a significant difference in the median yield between the acid and basic conditions with this small dataset.

Nevertheless, we can descriptively analyze and visualize the data conditions for acid and basic hydrolysis. See Figure 7. The box plot shown for the acid hydrolysis yield clearly demonstrates a higher median than the basic hydrolysis yield, which generally indicates a superior yield under acid conditions.

Analysis by FTIR-ATR Spectroscopy

The TPA obtained from the hydrolysis process was analyzed using Infrared Spectroscopy. The objective was to identify characteristic signals indicating the disappearance of the ester carbonyl bond and the emergence of a signal for the carboxylic acid group.

Consistent with similar research, these signals are expected to show a shift to lower wavenumbers for the C=O vibration of the carboxylic acid (1682 cm^{-1}) compared to the ester (1730 cm^{-1}). Additionally, the emergence of a broad band with overtones from 3300 to 2500 cm^{-1} is expected, corresponding to the O-H stretching vibration of a carboxylic acid (Ramírez y col., 2010; Sammon et al, 2000)

Figure 8 shows the IR spectra of the products obtained from acid (C) and basic (B) hydrolysis, as well as the IR spectrum of the collected PET (A).

Box 10

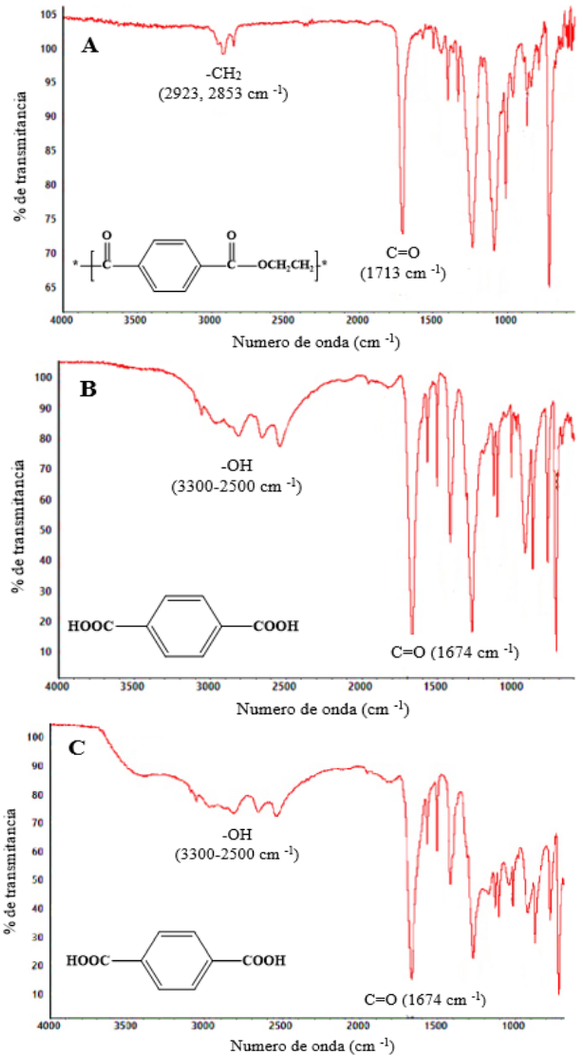

Figure 8
IR spectra of the collected PET sample (A), Basic hydrolysis product (B), and Acid hydrolysis product (C).

Source: Authors

In Spectrum A, the significant characteristic bands of PET are highlighted: the $-CH_2$ bond shows asymmetric stretching at 2923 cm^{-1} and symmetric stretching at 2853 cm^{-1}; the ester C=O stretching is found at 1713 cm^{-1}; the C=C stretching of the aromatic ring appears at 1408 cm^{-1}; and the in-plane C-H bending is observed in the region of 1241 to 1016 cm^{-1}.

In Spectra B and C, characteristic signals of TPA are shown. Both exhibit a broad band ranging from 3300 to 2500 cm^{-1}, corresponding to the O-H stretching vibration for acids; the C=O stretching vibration shifted to 1674 cm^{-1}; C=C stretching vibrations of the aromatic ring between 1574 and 1422 cm^{-1}; C-OH in-plane bending around 1423 cm^{-1}; and C-O stretching around 1279 and 1276 cm^{-1}.

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Conclusions

The collected PET, when subjected to acid and basic hydrolysis, yielded TPA as the final product, with average yields of 70.77% and 66.7%, respectively. Although the yields are very similar, the statistical analysis performed using the sign test found no significant difference between the medians of the yields from both treatments.

This indicates that, based on the available data, it cannot be asserted that one methodology is superior to the other in terms of yield. However, based on the experimental experience performing both methodologies, it can be stated that TPA from basic hydrolysis is obtained more easily and safely compared to TPA from acid hydrolysis. The products were confirmed through IR spectra, which highlighted characteristic bands showing the changes in functional groups from PET to TPA.

Obtaining TPA through chemical recycling is relevant from an environmental perspective, as it presents a distinct option for the treatment of plastic waste in El Salvador. Furthermore, this work can establish a precedent for potential chemical recycling feasibility studies in the country, offering an alternative to the mechanical recycling currently in practice.

Declarations

Conflict of interest

The authors declare no interest conflict. They have no known competing financial interests or personal relationships that could have appeared to influence the article reported in this article.

Author contribution

De la Cruz, Boris: Literature search, methodological design of basic hydrolysis, experimental process of basic hydrolysis, analysis of spectroscopic and yield data, and manuscript writing.

Domínguez, Ricardo: Literature search, methodological design of acid hydrolysis, experimental process of acid hydrolysis, analysis of spectroscopic and yield data, and manuscript writing.

Moreno, Juan: Literature search, methodological design of statistical analysis, application of statistics for data analysis, and manuscript writing.

Availability of data and materials

All data generated or analyzed during this study are included in this published article and its supplementary information files.

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Abbreviations

ATR	Attenuated Total Reflection
EG	Ethylene Glycol
FTIR	Fourier Transform Infrared Spectroscopy
HCl	Hydrochloric acid
H_3PO_4	Phosphoric acid
HNO_3	Nitric acid
H_2SO_4	Sulfuric acid
KOH	Hidróxido de potasio
NaOH	Potassium Hydroxide
NYL	Nylon
PE	Polyethylene
PET	Poly(ethylene terephthalate)
PP	Polypropylene
PS	Polystyrene
PVC	Polyvinyl Chloride
TPA	Terephthalic acid

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