International Journal for Multidisciplinary Research (IJFMR)
E-ISSN: 2582-2160 ● Website: www.ijfmr.com ● Email: editor@ijfmr.com

Synergistic Effects of Sulfuric Acid and Citric Acid in the Hydrolysis of Polyethylene Terephthalate Plastics

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ABSTRACT

This study investigated the hydrolysis of polyethylene terephthalate (PET) using sulfuric acid as the catalyst, with citric acid added as a potential modifier to improve terephthalic acid (TPA) yield and PET conversion due to its chelating and buffering properties. The research evaluated whether adding 10 mL of citric acid at varying concentrations (1 M, 3 M, and 5 M) could enhance hydrolysis efficacy compared to sulfuric acid alone, addressing inefficient PET hydrolysis and supporting plastic waste mitigation. Each setup used 5 g of PET in 100 mL of solution, including one control group with 80% sulfuric acid and three treatment groups with added citric acid. Hydrolysis was conducted at 100 °C for 30 minutes with constant stirring, followed by cooling, dilution, filtration, drying, and gravimetric analysis to determine TPA yield and PET conversion. Statistical analysis showed no significant difference in TPA yield between the sulfuric acid–only and citric acid–modified groups. However, significant differences were found in PET conversion, with post hoc results indicating superior conversion in the sulfuric acid–only group. Despite this, citric acid–treated samples produced more consistent TPA yields with lower variability and achieved near-complete conversion at higher concentrations. The sulfuric acid–only group showed highly variable yields, possibly due to increased viscosity and drying difficulty despite similar drying conditions, which may have affected statistical sensitivity. Overall, citric acid shows potential as a hydrolysis modifier, though further studies with improved drying protocols and larger sample sizes are recommended.

Keywords: Citric Acid, Hydrolysis, PET conversion, Sulfuric acid, Terephthalic acid

INTRODUCTION

Plastic pollution has become one of the most pressing environmental challenges worldwide due to the rapid increase in plastic production and the limitations of current waste management systems. Polyethylene terephthalate (PET) is widely used in beverage bottles and food packaging because of its durability, lightweight structure, and chemical resistance. However, these same properties make PET highly resistant to natural degradation, allowing large quantities of plastic waste to accumulate in landfills, waterways, and coastal environments. The environmental impact of PET waste is particularly evident in developing regions where recycling infrastructure remains limited. In the Philippines, PET bottles and other single-use plastics contribute significantly to the country's solid waste problem, creating long-term environmental concerns for both terrestrial and marine ecosystems (DENR, 2024). Because of these challenges, researchers have increasingly explored chemical recycling processes that can recover valuable materials from plastic waste instead of relying solely on mechanical recycling or disposal methods. One promising approach to PET recycling is chemical depolymerization through hydrolysis, which converts PET back into its monomer components such as terephthalic acid (TPA). Acidic hydrolysis has been identified as an effective process for producing high-purity TPA, although it often requires highly concentrated acids and specific reaction conditions to achieve optimal yields (Siddiqui et al., 2020; Damayanti & Wu, 2021).

Studies have shown that while strong acid catalysts such as sulfuric acid can facilitate the breakdown of PET polymer chains, several challenges remain in maximizing the recovery of TPA. These challenges include oxidative effects, carbonization of ethylene glycol, and degradation of TPA during the reaction process, which can significantly reduce the overall yield (Ügdüler et al., 2020; Cao et al., 2022). In addition, reactions conducted below the melting point of PET often require longer reaction times and strict conditions, making the process less efficient for potential large-scale applications (Cao et al., 2022). These limitations highlight the need for further investigation into reaction conditions and catalyst combinations that can improve the efficiency of PET depolymerization. In the Philippines, the limited amount of local research focusing on advanced depolymerization techniques indicates a gap in scientific efforts aimed at recovering high-purity terephthalic acid from PET waste (DOST-PTRI, 2024). This gap restricts the country's progress toward developing a circular plastic economy and contributes to continued dependence on virgin PET production (UNDP Philippines, 2024). A study has shown that citric acid combined with dimethyl sulfoxide improved TPA extraction in PET hydrolysis, but was conducted in a different approach wherein they focused on alkaline hydrolysis and not acid hydrolysis (Nguyen & Chiang, 2024).

Moreover, despite there being studies showcasing the positive effects of citric acid in combination with another acid in other applications, there remains a lack of studies investigating the combined use of citric acid with sulfuric acid during the hydrolysis process, despite the potential of such catalyst systems to influence reaction efficiency and product yield (Kibria et al., 2023). Addressing this gap is particularly relevant in regions such as Davao Oriental, where plastic waste pollution continues to affect coastal and marine environments (Verzosa et al., 2024). Therefore, the objectives of this study are to determine the yield of terephthalic acid recovered from PET waste through hydrolysis and to evaluate the effectiveness of selected experimental conditions in maximizing recovery efficiency. By establishing a controlled laboratory-scale procedure and assessing the consistency of the resulting TPA yield with theoretical expectations, this study aims to contribute to improving the reliability of chemical recycling strategies and support sustainable plastic waste management practices.

RESEARCH QUESTIONS

1. Is there a significant difference between the TPA yield of the sulfuric acid solution in an 80% v/v concentration and its overall efficacy in comparison to the combination of sulfuric and citric acid solution during hydrolysis?

2. Does the combined acid solution reduce the amount of unwanted byproducts, such as the unreacted PET fragments during hydrolysis?

METHODS

Study design

This study employed a quantitative research design by Creswell, W. John; Creswell. (2022) involving the process of collecting, analyzing, and interpreting the data, which also involved writing the possible results of the study. This study measured the dependent, the synergy of sulfuric and citric acid and independent variable, Terephthalic Acid and the amount of unwanted byproducts. The quantitative data from the dependent variable was obtained from the adapted formula of Islam et al. (2023). This study also employed an experimental method wherein the researchers assess the variable. The data is collected on an instrument that measures the reaction of the variable, and the information is analyzed using statistical procedures and hypothesis testing. Specifically, the experiment involved controlled conditions to isolate the impact of the sulfuric-citric acid solution. This rigorous approach ensured the reliability and validity of the findings regarding the synergistic effects. This method of research gave insights and allowed the researchers to determine its synergistic effects in the hydrolysis of PET plastics and TPA yield of the sulfuric-citric acid solution compared to the sulfuric solution alone.

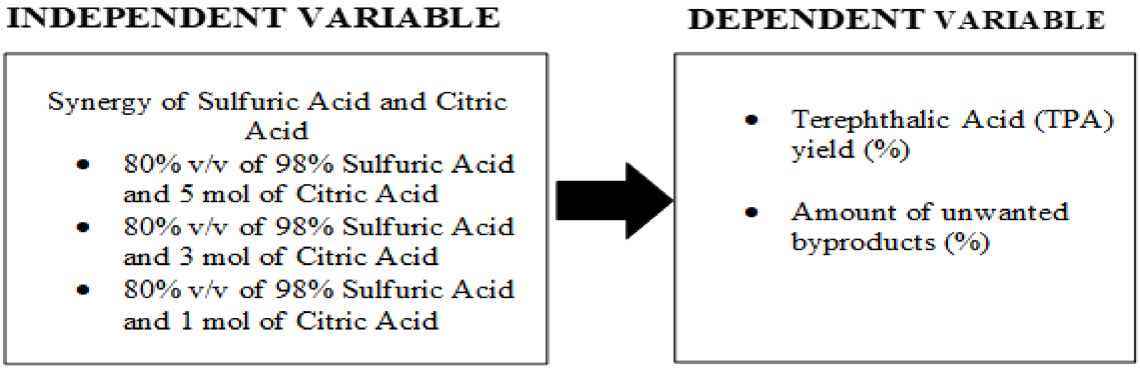
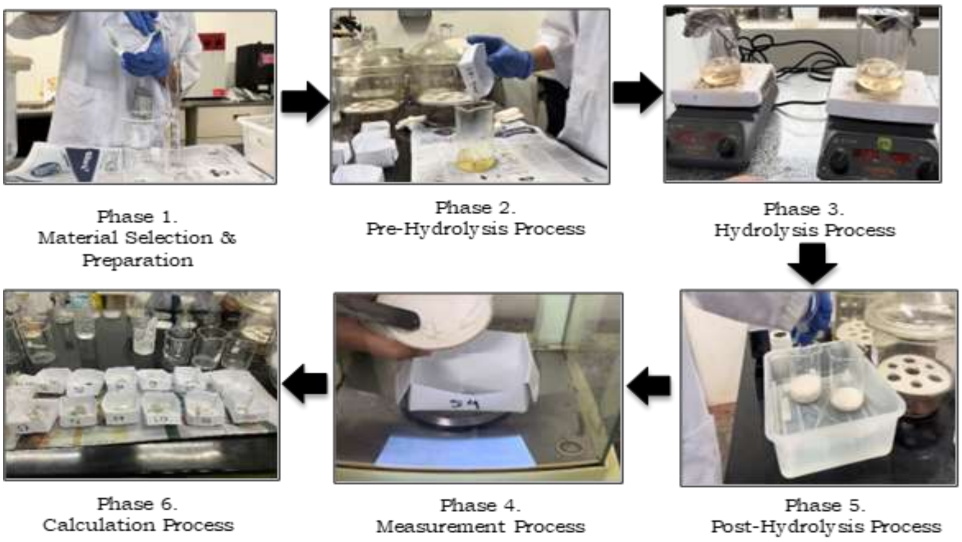


Figure 1. The Conceptual Framework of the Study.

Instrumentation


Phase 1. Material Selection & Preparation

Phase 2. Pre-Hydrolysis Process

Phase 3. Hydrolysis Process

Phase 4. Measurement Process

Phase 5. Post-Hydrolysis Process

Phase 6. Calculation Process

Figure 2. Data Collection Procedure

The researchers prepared hydrolysis liquor for each experimental set at 80% v/v acid concentration, except Set A which used only water. Set B used diluted sulfuric acid, while Sets C, D, and E included sulfuric acid combined with citric acid solutions of 1 M, 3 M, and 5 M. The citric acid solutions were prepared by dissolving measured amounts of citric acid monohydrate in distilled water to achieve the required molarity. Appropriate volumes of sulfuric acid, citric acid solution, and distilled water were then mixed to obtain the final hydrolysis liquor for each treatment set.

The next procedure of five treatment sets were prepared: Set A used water only, Set B used sulfuric acid only, while Sets C, D, and E used sulfuric acid combined with 1 M, 3 M, and 5 M citric acid, respectively. Each set contained three samples with a 1:20 ratio of PET to hydrolysis liquor (5 g PET to 100 ml solution). The initial weight of PET and chemicals used were recorded before proceeding with hydrolysis.

Followed by five sets containing 5 g PET and their respective hydrolysis liquor were placed in flat-bottom flasks and partially covered with aluminum foil to reduce liquor loss. The flasks were heated on a hot plate at 100 °C while stirring at 300 rpm using a PTFE-coated magnetic stirrer. Once the solution reached 100 °C, the reaction was maintained for 30 minutes to facilitate the hydrolysis of PET.

After hydrolysis, the solutions were cooled to room temperature and diluted with an equal amount of distilled water to allow TPA to precipitate. The solids were filtered using Whatman No.1 filter paper, separating TPA from unreacted PET fragments. The TPA was washed twice with cold distilled water, dried in an oven at 105 °C for 2 hours, and stored in a sealed container. The remaining filtrate was neutralized with baking soda before disposal.

Afterwards, the dried TPA was weighed using an analytical balance at Davao Oriental State University (DOrSU) to obtain accurate measurements. The unreacted PET fragments were also weighed to determine the remaining material after hydrolysis.

The researchers calculated the efficiency of the hydrolysis by determining the conversion of PET and the yield of terephthalic acid (TPA). This involved computing the moles of TPA produced, estimating the theoretical amount of TPA that could be obtained from the PET used, and using these values to determine the overall yield and conversion efficiency of the process.

Data Analysis

The raw experimental data, including the initial mass of PET, mass of recovered terephthalic acid (TPA), and mass of unreacted PET plastics, were used to compute TPA yield (%) and PET conversion (%) following the formula adapted from Islam et al. (2023), and these calculated percentages served as the derived variables for statistical analysis. The data were organized using Microsoft Excel prior to statistical treatment. Given the small sample size per treatment group ($n = 3$) and the absence of normality assumptions, non-parametric methods were employed; specifically, the Kruskal–Wallis H test was used to determine whether significant differences existed among the five treatment sets, particularly between the sulfuric acid–only positive control and the groups with varying citric acid concentrations. When significant differences were identified, Dunn’s post hoc test was conducted to determine which specific treatment groups differed. Median values were also examined to identify observable patterns or trends across treatments. All data, including values considered outliers or those potentially influenced by incomplete drying, were retained to avoid data exclusion bias since all samples underwent identical drying conditions, and statistical computations were performed by a professional statistician.

RESULTS

Table 1 summarized the percentage yield of TPA for Set A (control), Set B treated with 80% (v/v) H_2SO_4, and Sets C–E treated with 70 mL of 98% H_2SO_4 combined with 10 mL of citric acid at concentrations of 1 M, 3 M, and 5 M, respectively.

Table 1. TPA Yield in Percentage

	SET A (Control)	SET B (80% v/v H_2SO_4)	SET C (70 ml H_2SO_4 (98%) + 10 ml 1M citric acid)	SET D (70 ml H_2SO_4 (98%) + 10 ml 3M citric acid)	SET E (70 ml H_2SO_4 (98%) + 10 ml 5M citric acid)
Sample 1	0%	121.13%	38.39%	66.97%	46.07%
Sample 2	0%	22.59%	61.34%	62.17%	87.93%
Sample 3	0%	164.20%	32.36%	136.39%	78.16%

The control (Set A) showed 0% yield and conversion, confirming ineffective hydrolysis without the presence of a catalyst. Set B (80% v/v sulfuric acid) performed best, reaching up to 164.20% TPA yield. Among combined-acid sets, Set C gave the lowest yields, likely due to low citric acid moles. Set D showed higher yields but included an outlier above 100%. Set E (with 5M citric acid) produced the most consistent and reliable yields reaching near 100%.

Table 2 showed Conversion of PET Plastic in Percentage (%) for Set A (control), Set B treated with 80% (v/v) H_2SO_4, and Sets C–E treated with 70 mL of 98% H_2SO_4 combined with 10 mL of citric acid at concentrations of 1 M, 3 M, and 5 M, respectively.

Table 2. Conversion of PET Plastic in Percentage (%)

	SET A (Control)	SET B (80% v/v H_2SO_4)	SET C (70 mL H_2SO_4 (98%) + 10 mL 1M citric acid)	SET D (70 mL H_2SO_4 (98%) + 10 mL 3M citric acid)	SET E (70 mL H_2SO_4 (98%) + 10 mL 5M citric acid)
Sample 1	0%	100%	9.58%	33.84%	56.06%
Sample 2	0%	100%	40.77%	70.59%	80.13%
Sample 3	0%	100%	34.66%	90.81%	97.54%

Set C had the lowest conversions (9.80–40.77%), indicating more unreacted PET. Set D showed higher conversion (33.84–90.81%), meaning less unreacted plastic. Set E achieved the highest and most consistent conversion (46.07–87.93%), leaving the least unreacted PET overall.

Table 3 showed the median values for each test set, the middle value of three samples measures for TPA yield and PET conversion.

Table 3. Median of TPA yield and Conversion of PET plastic

SET	Citric Acid (Mol)	TPA Yield (%) Median	PET conversion (%) Median
A	0	0%	0%

B	0 ((H_2SO_4 only)	121.13%	100%
C	1	38.39%	34.66%
D	3	66.97%	70.59%
E	5	78.16%	80.13%

For Set A, both the TPA yield and PET conversion were 0%, showing that PET did not break down without an acid catalyst. In SET B, the median TPA yield was 121.13%, and the median PET conversion was 100%, which means sulfuric acid alone worked very well. When citric acid was added to SETS C to E, the samples gradually improved.

Table 4 represented the Kruskal–Wallis test results comparing TPA yield (%) among treatment groups with varying citric acid concentrations. The table reported the Kruskal–Wallis H statistic and its corresponding p-value.

Table 4. Kruskal–Wallis Test for TPA Yield (%)

Statistic	**Value**
H-value	2.21
p-value	0.529

The Kruskal–Wallis test for TPA yield (%) (H = 2.21, p = 0.529) indicated no statistically significant difference in TPA yield among the different acid treatments due to the p value being greater than 0.05.

Table 5 represented the Kruskal–Wallis test results comparing PET Conversion (%) among treatment groups with varying citric acid concentrations. The table reported the Kruskal–Wallis H statistic and its corresponding p-value.

Table 5. Kruskal–Wallis Test for PET Conversion (%)

Statistic	**Value**
H-value	9.78
p-value	0.021

The Kruskal-Wallis test for PET Conversion (%) indicated that there was a statistically significant difference among the different acid treatments (H = 9.78, p = 0.021). This suggested that one or two of the treatments exhibited a significant difference against the other treatments. Thus, Dunn's Post-Hoc Test was conducted to determine the specific treatment responsible for this.

Table 6 represented Dunn's post-hoc pairwise comparisons for PET conversion (%) following a significant Kruskal–Wallis test. Adjusted p-values indicated statistically significant differences between specific treatment groups.

Table 6. Dunn's Post-Hoc Test for PET Conversion (%)

Comparison	Adjusted p-value	Interpretation
H_2SO_4 vs H_2SO_4 + 1 M citric	< 0.05	Significant
H_2SO_4 vs H_2SO_4 + 3 M citric	< 0.05	Significant
H_2SO_4 vs H_2SO_4 + 5 M citric	< 0.05	Significant

Dunn's Post-Hoc test for PET Conversion (%) indicated that all combined treatments showed significantly lower PET conversion (%) compared to Sulfuric Acid alone. This suggested that adding citric acid, even at increasing concentrations (1–5 M), inhibited the depolymerization efficacy of sulfuric acid, either due to dilution or interference with the acidic hydrolysis process.

DICSUSSION

This study evaluated the effect of increasing citric acid concentration in sulfuric acid–catalyzed PET hydrolysis by analyzing TPA yield and PET conversion. While statistical testing showed no significant difference in TPA yield among treatments ($H = 2.21$, $p = 0.529$), a significant difference was observed in PET conversion ($H = 9.78$, $p = 0.021$), with sulfuric acid alone achieving the highest conversion, consistent with Panjaitan et al. (2023) on its strong ester bond cleavage capability. It is importance to highlight that despite the lack of statistical significance in TPA yield, a clear upward trend was observed: as citric acid concentration increased, median TPA yield improved and variability decreased, indicating enhanced reaction stability and more consistent outcomes. This pattern suggests a concentration-dependent effect of citric acid that may not have reached statistical significance due to experimental constraints. Barredo et al. (2023) noted that hydrolysis efficiency is highly dependent on reaction time and conditions, and the short duration (30 min at 100 °C) may have limited measurable yield differences. Extreme variability in the sulfuric acid–only group for TPA yield (%), partly due to gravimetric sensitivity to residual moisture as discussed by Islam et al. (2023), including yields exceeding 100% from incomplete drying given the current conditions (105 °C for 2 hours), likely reduced statistical power in the Kruskal–Wallis test but were included to prevent data exclusion since all samples received equal drying conditions. Although combined acid systems showed slightly lower overall conversion than sulfuric acid alone, near-complete conversions (90.81% and 97.54%) were achieved at higher citric acid levels, supporting reports of potential synergistic effects under optimized conditions (Nguyen & Chiang, 2024; Roshanfar et al., 2024). Consistent with Worku et al. (2023), the observed improvement in yield consistency at higher citric acid concentrations suggests that combining strong and weak acids may enhance catalytic stability. However, the small sample size ($n = 3$), short reaction time, and insufficient drying time limited statistical sensitivity (Cao et al., 2022; Creswell & Creswell, 2022). Overall, sulfuric acid remained the dominant catalyst under the tested conditions, yet the progressive improvement in yield and stability with increasing citric acid concentration indicates promising system-dependent enhancement that warrants further optimization of reaction conditions.

CONCLUSION

The production of terephthalic acid (TPA) was influenced by the acid treatment applied during PET hydrolysis. Sulfuric acid produced higher TPA yield, while the combination of sulfuric and citric acids was still able to generate TPA but with lower efficiency. Variations in experimental results affected the reliability of certain treatment sets, indicating that measurement outcomes were strongly influenced by experimental conditions. Overall, the synergy of acids demonstrated potential, although sulfuric acid alone remained more effective.

Based on the results of the study, the following recommendations are summarized: Future studies may further examine the effectiveness of acid combinations in PET hydrolysis. Researchers may improve experimental conditions to obtain more consistent and reliable results. Further investigations may focus on optimizing the use of acid synergy while maintaining effectiveness in TPA production.

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