

Research Article

Efficient Bioethanol Production from Yam Peel Waste via Acid Hydrolysis and Fermentation

Kosi Mawuena Novidzro^{1,*} , Gnimdou Issanga Abli¹ , Sassou Megnassan² ,
Kossi Honore Koumaglo¹ 

¹Department of Chemistry, University of Lomé Lomé Togo

²Department of Chemistry, University of Kara, Kara, Togo

Abstract

Food residues can be transformed into bioethanol, reducing CO₂ and methane emissions while fostering sustainable development. This method provides a cost-effective way to enhance the value of non-edible food sources. Thus, yam peels produced as agro-industrial waste is perfect starting material for bioethanol production. The main goal of this study is to evaluate the efficiency of bioethanol production from yam peels. Thus, fresh peels are subjected to wet milling to obtain a starch-rich powder. The hydrolysis of dry extracts, optimized according to time, acid concentration, and dry extract (DE)/water volume ratio, is carried out by reflux heating in the presence of different concentrations of H₂SO₄ used as a catalyst. The ethanolic fermentation of the hydrolysate musts, after adjusting the pH to 4.5, is conducted in batch mode using *Saccharomyces cerevisiae*. Fermentation monitoring is ensured by measuring Brix with an Abbe refractometer, while the ethanol content is determined by the pycnometric method, in accordance with the recommendations of the Association of Official Analytical Chemists (AOAC). According to the results obtained, the optimal hydrolysis conditions include: an H₂SO₄ concentration of 8% (w/w), a dry extract (DE)/water volume ratio of 1:5 (g/mL), and a duration of 2 hours. These conditions yield an ethanol content of 6.72 ± 0.26% (v/v), corresponding to 264.97 ± 10.07 g EtOH/kg of dry matter. Wet milling provides better ethanol yields compared to dry milling. Finally, bioethanol production from yam peels prevents their degradation into CO₂ and methane, which are greenhouse gases. The results from this study are important for the commercial production of bioethanol through a process of valorizing plant resources and reducing waste to promote the circular economy.

Keywords

Yam Peels, Acid Hydrolysis, Fermentation, Bioethanol, Environmental Benefit

1. Introduction

The valorization of agricultural residues has become a major issue in strengthening sustainable development worldwide, particularly in regions with high agri-food production. Yam is a staple crop cultivated in almost all five

regions of Togo. According to the Department of Agricultural Statistics, Informatics and Documentation (DASID), national production of yam for the 2020-2021 season in Togo reached 940,876 tons [1]. Considered a staple food for about half of

*Corresponding author: donnenovi@yahoo.fr (Kosi Mawuena Novidzro), donnenovi@gmail.com (Kosi Mawuena Novidzro)

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the Togolese population, yam is consumed in various forms, namely boiled, roasted, fried, pounded, or as flour paste. It stands out due to its higher protein content and its higher calorie value than cassava [2, 3].

Before being prepared as food for consumption, yam tubers first undergo an initial mechanical pretreatment known as peeling. During this preliminary step, the peels, also referred to as yam skins, are generated as waste or food residues. Traditionally, these peels are used to feed animals. However, there is currently growing interest in their valorization. Consequently, bioethanol is one of the high-value products that is produced by a series of intricate reactions carried out on a biorefinery platform [4].

Thus, once limited to self-consumption, yam has now become a cash crop that produces starch, a white flour rich in starch used to make glucose and cakes, or to prepare dishes such as “*foufou*” and other industrial products [5]. Today, the benefits of yam are also exploited in the pharmaceutical and cosmetic industries for the production of medicines against skin diseases [3, 6]. Furthermore, comparative studies show that agricultural residues, including yam peels, can be valorized for bioethanol production through suitable fermentation processes [7, 8]. Since the fermentation of cassava peels has provided really interesting yields, this also suggests a similarly favorable potential for yam [9].

Among the most commonly cultivated yam varieties in Togo, there are traditional varieties such as “*Kratchi*”, “*Florido*”, “*Hè-abalo*”, “*Alassora*”, “*Laboco*”, and “*Gnamiti*”, as well as improved varieties such as TDR 99/01169, TDA 89/02665, “*Brutani*”, “*Motchi*”, and “*Moniya*” [1]. With their considerable starch content, yam peels could serve as a basic raw material for the bioethanol production. Bioethanol use can contribute to reducing greenhouse gas emissions and limiting dependence on fossil fuels [7, 8, 10]. The existential rivalry between food crops and those used for energy production may be avoided by using agricultural wastes, such as yam peels, to produce Bioethanol. This will reduce criticisms of first-generation biofuels. [10-12].

The process of converting yam peels into bioethanol involves several steps: biomass pretreatment, hydrolysis - either enzymatic or acid - to release fermentable sugars, and finally, the fermentation of these sugars, usually realized with yeasts of the genus *Saccharomyces cerevisiae*. To maximize the yield of bioethanol, the conditions of each step involved in the process must be optimized [13]. These practices promote the shift to renewable energy sources and align with the circular economy paradigm because all agri-food byproducts may be repurposed to produce new add-value product. The conversion of yam peels into bioethanol could also contribute to diversifying income sources for local populations, particularly in rural areas. Consequently, this valorization improves the well-being of local communities and strengthens the sustainability of yam cultivation.

In order to provide a sustainable substitute for fossil fuels in a world striving for a circular economy, this study focuses on

evaluating the effectiveness of bioethanol production from yam peels by investigating appropriate pretreatment procedures and ideal ethanol fermentation methods.

2. Materials and Methods

2.1. Materials Used for Bioethanol Production

Yam peels, by-products from yam (*Dioscorea* spp.) processing, constitute a biomass rich in starch and fermentable sugars. Often regarded as waste, they nonetheless represent a valuable resource for industrial applications, particularly for bioethanol production. In this study, yam peels (Figure 1) used as raw material for bioethanol production, were collected in 2023 from households and restaurants in Agoè a city located in the northern suburbs of Lomé the capital of Togo.



Figure 1. Picture of yam peels used as raw material.

2.2. Methods

2.2.1. Experimental Design for Bioethanol Production from Yam Peels

Two pretreatments were applied with the yam peels such as air drying followed by milling to obtain a fine particle size, and wet milling intended to extract a starch-rich powder. These two methods aim to prepare effectively the samples for their conversion into bioethanol. When brought back to the laboratory, the yam peels were mechanically milled using a blender to obtain the flour of a starch-rich powder. Then, this flour was hydrolyzed in an aqueous medium by homogeneous acid catalysis. The acids used as catalysts for this step included HCl, H₂SO₄, HNO₃, and H₃PO₄. The parameters investigated for acidic hydrolysis were type of acid pre-treatment and acid content (0%; 1%, 2%, 4%, 6%, 8%, 10%, and 12%: v/v). A sample without any acid pre-treatment was also prepared and used as a control [14].

For each sample, the flour was mixed with water and one of the selected acids. Then, the mixture was heated under reflux to ensure the depolymerization of the polysaccharides which derived from the flour. Thus, the hydrolysates obtained were

filtered through filter paper, producing syrups. Before proceeding to the crucial step of ethanolic fermentation, the pH of the syrups was adjusted to 4.5 using a normal caustic soda solution [15]. Then, the process so-called “Separate Saccharification and Fermentation” (SSF) process was carried out under anaerobic conditions [16].

2.2.2. Physicochemical Characterization of Yam Peels

The physicochemical properties of the flour of yam peels were evaluated by the determinations of the water and volatile matter content (WVC), the dry matter ratio (DMr), and the ash content (Ac) [15]. The formula (1), formula (2) and formula (3), were used respectively for these determinations [17, 18].

$$WVC = \frac{MP - MD}{MP - ME} \times 100 \quad (1)$$

$$WVC + DMr = 100\% \quad (2)$$

$$Ac = \frac{M_1 - M_0}{M_2 - M_0} \times 100 \quad (3)$$

With:

MP = mass of the flour of yam peels + Petri dish before being placed in the oven;

MD = mass of dried flour of yam peels + Petri dish after being placed in the oven;

ME = mass of the empty Petri dish.

Ac = Ash content of the flour of yam peels;

M₀ = Mass of the empty crucible;

M₂ = Total mass of the crucible and the flour of yam peels before incineration;

M₁ = Total mass of the crucible and the ash after incineration.

2.2.3. Wet Milling Pretreatment of Peels

Another pretreatment applied with the yam peels consisted of wet milling by adding water before milling. In this method, a water/yam peel ratio of 2:5 was used to facilitate and increase the starch extraction yield. After sieving and decantation, the solid residue obtained by wet milling, deposited at the bottom of the container, was dried to obtain a powder particularly rich in starch [17]. This wet milling and decantation processes were used to reduce the size of the polymers present in the flour of yam peels. Thereby, this process allowed to increase the contact surface between the polymers and the catalyst, which facilitates their depolymerization [19]. In addition, this process helped to promote the release of fermentable sugars necessary for bioethanol production.

2.2.4. Hydrolysis of Polysaccharides Contained in the Flour of the Yam Peels

The hot hydrolysis method by homogeneous acid catalysis with H₂SO₄ was adopted in the current study due to its rela-

tively low cost compared to enzymatic hydrolysis, and due to its swiftness [20]. Nevertheless, its drawbacks such as the formation of inhibitory by-products including furan compounds and organic acids, can interfere with subsequent fermentation step [21]. In addition, the use of strong acids in hydrolysis step leads to increased equipment corrosion, requiring resistant and costly materials [22]. Finally, the treatment of acidic effluents, a necessary step to neutralize acid residues, increases the complexity and environmental impact of the process [23].

The conditions of the acid hydrolysis were optimized using an experimental method known as the monothetic analysis which referred to in English as One Factor at a Time (OFT). This approach consists of varying only one parameter at a time. The other parameters remain constant, thereby allowing the effect of each variable to be evaluated independently. Thus, three key factors influencing the hydrolysis reaction with H₂SO₄ catalyst were investigated. These include H₂SO₄ content, the hydrolysis time, and the flour/water ratio used. To optimize the hydrolysis, the acid content has been varied between 0% and 12% (w/v) and the dry flour/acidified water volume ratios tested were 1:10, 1:5, 3:10, and 2:5. The final volume of each heterogeneous liquid mixture prepared for acid hydrolysis was 500 mL. The hydrolysis reaction was carried out by reflux heating in a Pyrex flask with a capacity of 1 L. To continuously homogenize the mixture, the pumice stones were added to the solutions to ensure uniform boiling. The solutions were heated under reflux for durations ranging from 1 to 6 hours, namely 1 h, 2 h, 4 h, and 6 h, according to the experimental conditions selected after a few preliminary trials.

After the acid hydrolysis step, the hydrolysates were cooled to room temperature. Then, they were filtered to remove the solid residues they contained. Then, the filtrates were collected in tightly sealed flasks and stored at -23 °C for future use, particularly as fermentable musts for bioethanol production.

2.2.5. Ethanolic Fermentation Method Applied and Bioethanol Purification

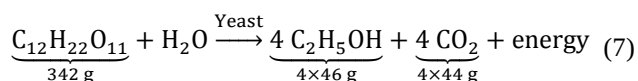
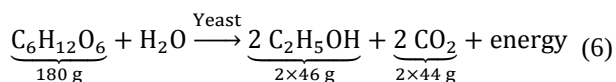
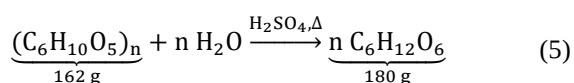
In this study, the ethanol production was carried out using batch fermentation [24]. This fermentation process was conducted at ambient temperature (30 °C) for 120 hours (5 days) with 1 g/L of yeast at the beginning. This process was catalyzed by active dry yeast of the genus *Saccharomyces cerevisiae* (Saf-Levure S. I. Lesaffre), used to convert the fermentable sugars present in the syrups into bioethanol.

At the final stage, the bioethanol produced was recovered and purified by a distillation method, using complete distillation equipment [16]. This equipment was consisted of a heating mantle, a round-bottom flask of 1,000 mL, a Vigreux column, a condenser, as well as a volumetric flask 500 mL for the distillate's collecting.

2.2.6. Bioethanol Production Yields

The theoretical yield, known as the Gay-Lussac yield (GLY), for bioethanol production from substrates such as starch or sucrose, was calculated using the balanced equations of the key chemical reactions. For starch, the theoretical yield (Formula (4)) is based on the balanced equation of acid saccharification leading to the formation of glucose (Equation (5)), followed by the equation describing the ethanolic fermentation of glucose into ethanol (Equation (6)). The balanced equation of ethanolic fermentation of sucrose into ethanol (Equation (7)), calculated using Formula (8).

$$\text{GLY (\%)} = \frac{2 \times 46 \text{ g EtOH}}{162 \text{ g Starch}} = 0,5679 \text{ g EtOH/g Starch} \quad (4)$$



$$\text{GLY (\%)} = \frac{4 \times 46 \text{ g EtOH}}{342 \text{ g Saccharose}} = 0,538 \text{ g EtOH/g Saccharose} \quad (8)$$

2.2.7. Data Analysis

For every experiment, three trials were conducted, and the average of the three trials was taken into account. The results are presented as mean (M) $\pm$ Standard Error of the Mean (SEM).

3. Results and Discussion

3.1. Physicochemical Characteristics of Yam Peels

The physicochemical characteristics of yam peels, including their water and volatile matter content (WVC), dry matter ratio (DMr), mineral matter or ash content (Ac), as well as their organic matter content (OMc), are illustrated in the form of a two-dimensional pie chart (Figure 2).

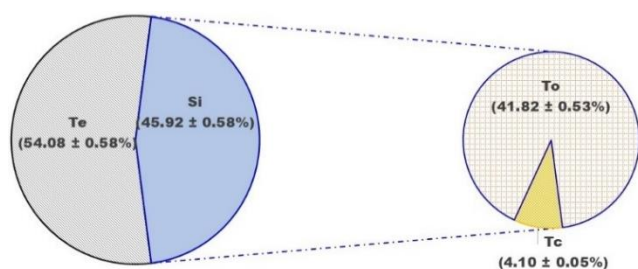


Figure 2. Physicochemical characteristics of yam peels.

The interpretation of the data from Figure 2 reveals that yam peels have a water and volatile matter content (WVC) of $54.08 \pm 0.58\%$. In addition, the dry fraction, corresponding to a dry matter ratio (DMr) of $45.92 \pm 0.58\%$, is divided into an ash content (Ac) of $4.10 \pm 0.05\%$ and an organic matter fraction (OMc) of $41.82 \pm 0.53\%$. Yam peels' physicochemical properties showed that this kind of waste was high in organic content, including a biodegradable fraction that can be converted to bioethanol. This organic matter content ($45.92 \pm 0.58\%$) was far higher than the 28.79% found in the previously published study on cashew apples by [15], which produced bioethanol with very promising results. The ash content obtained ($4.10 \pm 0.05\%$) in this study for yam peels is slightly lower than the values of $5.5 \pm 0.5\%$ and $5.75 \pm 0.25\%$, respectively found by [18] for papaya and banana peels used in bioethanol production. However, the water and volatile matter content of yam peels ($54.08 \pm 0.58\%$) was very lower than those of papaya and banana peels ($89.42 \pm 0.92\%$ and $89.42 \pm 0.58\%$, respectively) [18]. Furthermore, the work reported by [25] on bioethanol production with Watermelon (*Citrullus lanatus*) revealed a water and volatile matter content of $93.11 \pm 1.27\%$ and an ash content of $0.92 \pm 0.46\%$. A water and volatile matter content of 14% and a dry matter ratio of 86% - which equated to mineral contents (or ashes) of 5% and organic matter of 81% - were reported by [26] in the study published on the conversion of pineapple peels into bioethanol production.

3.2. Comparative Analysis of the Two Processes Used for Yam Peel Milling

In Table 1 are displayed the concentrations in terms of total soluble solids TSS (°Brix) of the two hydrolysates produced following the hydrolysis of the extracts from the two methods of peel milling, as measured.

Table 1. Influence of the milling process on the concentration of yam peel hydrolysates.

Milling process	TSS (°Brix)
Dry milling	13.25 ± 0.14
Wet milling	15.17 ± 0.17

In this study, the wet milling of yam peels resulted in a hydrolysate with a concentration of 15.17 ± 0.17 °Brix, higher than the 13.25 ± 0.14 °Brix obtained for dry milling. Thus, the wet milling is more efficient than dry milling for extracting starch granules. These findings are in agreement with the research done by some authors who highlighted that wet milling increases the yield of starch extraction because the grain's cells are softer after being soaked in water, which makes it easier to separate the constituent parts mechanically. Compared to dry approaches, this technique improves starch

granule release and purifying efficiency, leading to a more thorough extraction [27].

3.3. Effect of H₂SO₄ Concentration on the Ethanol Production Parameters from Yam Peels

In Table 2 is shown the total soluble solids (TSS), Final Attenuation (FA); the Ethanol Content (EC); and the Ethanol Yield (EY) of H₂SO₄ hydrolysates after two hours of the hydrolysis of starchy extract with solid-liquid (S-L) ratio of 1:5. The data presented in Table 2 indicated that H₂SO₄ contents less than 2% are insufficient for a complete hydrolysis reaction of the starch. Thus, reflux heating of the corresponding aqueous mixtures at these concentrations after two hours have led to the formation of a viscous suspension or a

pasty solid known as starch paste, which is not fermentable into bioethanol. This starch paste formation could be explained by the insufficient amount of H₃O⁺ ions required to break down all the glycosidic bonds contained in the polymer chains contained in the starch extract. However, with H₂SO₄ content above 2%, reflux heating of an aqueous starch extract mixture with a S-L ratio of 1:5 after two hours results in a syrup, which TSS increased from 12.83 ± 0.44% to around 15.33 ± 0.44% using H₂SO₄ concentration of 12%. Compared to the different H₂SO₄ concentrations tested in this study, 8% is the best H₂SO₄ concentration to achieve optimal hydrolysis of the starch extract from yam peels. This conclusion is reinforced by the importance of minimizing the formation of inhibitory by-products, such as furfurals, often associated with high acid contents [28].

Table 2. Comparison of H₂SO₄ concentration on the ethanol production from yam peels.

H ₂ SO ₄ (%)	Hydrolysate Types	TSS (°Brix)	FA (%)	EC (%: v/v)	EY (%)
0	Paste	0.00	0.00	0.00	0.00
1	Paste	0.00	0.00	0.00	0.00
2	Syrup	12.83 ± 0.44	9.52 ± 2.24	0.18	1.23 ± 0.82
4	Syrup	14.00 ± 0.00	18.56 ± 2.92	0.18 ± 0.12	12.97 ± 0.95
6	Syrup	14.67 ± 0.33	35.59 ± 1.17	1.87 ± 0.14	28.62 ± 0.89
8	Syrup	15.33 ± 0.44	43.34 ± 0.91	4.12 ± 0.13	39.98 ± 2.28
10	Syrup	15.33 ± 0.44	46.30 ± 0.57	5.76 ± 0.33	39.95 ± 0.88
12	Syrup	15.67 ± 0.33	48.45 ± 0.03	5.75 ± 0.13	43.09 ± 2.85

TSS: Total Soluble Solids; FA: Final Attenuation; EC: Ethanol Content; EY: Experimental Yield

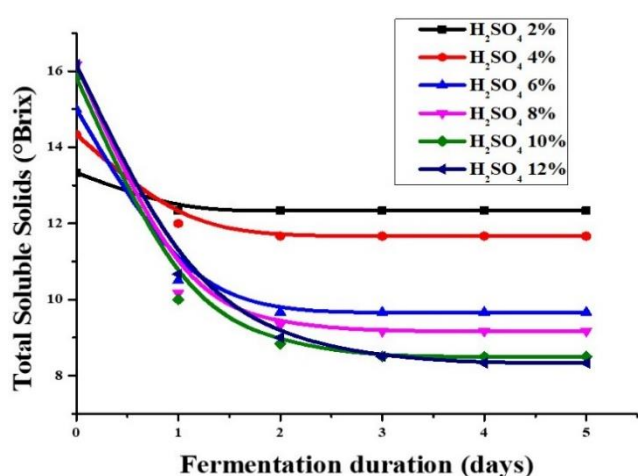


Figure 3. TSS variation of the hydrolysate musts as a function of H₂SO₄ concentration used.

The results presented in Figure 3 show the evolution of the total soluble matter content during the process of the batch ethanolic fermentation of the different hydrolysate musts from yam peels, depending on the H₂SO₄ concentration used.

The data from the Figure 3 were used to determine the final attenuation (FA) after the end of the must fermentation. The fermentation results indicated that the highest FA of 48.45 ± 0.03% is obtained with 12% H₂SO₄. In general, the ethanol production by fermentation, using yeast *Saccharomyces cerevisiae*, leads to a high yield when the FA exceeds at least 50% [26]. Taking this consideration into account, the optimal H₂SO₄ content for achieving better ethanol fermentation of yam peels ranges between 8% and 12%, as shown.

The data shown in Table 2 indicated that the H₂SO₄ concentration significantly influences the ethanol content (EC), ranging from 0.18 ± 0.12% (v/v) to 6.21 ± 0.71% (v/v). The highest EC values were achieved with 8%, 10%, and 12% of H₂SO₄. From the EC values determined, the experimental yields (EY) of bioethanol production are calculated and the values obtained are shown in Table 2. According to these

findings, 244.72 ± 16.19 kg of pure ethanol may be produced from one ton of yam peels, using 12% H_2SO_4 .

3.4. Statistical Analysis of the EC Results

The statistical analysis of the EC using the *one-way* ANOVA test (Table 3) indicates that there are significant differences between the ethanol contents produced in the musts as a function of the H_2SO_4 content applied (p -value < 0.0001). In particular, the use of 12% H_2SO_4 yielded the fermented must with the highest bioethanol content, suggesting that the optimal H_2SO_4 content for the hydrolysis of yam peel polymers is 12%.

Table 3. Summary of the *one-way* ANOVA test in the case of variation of H_2SO_4 concentration and their SD.

Parameters	Values
F	105.6
P-value	0.0001
SD ($P < 0.05$)	Yes
R-squared (R^2)	0.9778

SD: Significant difference

Table 4. Comparison of mean differences between various H_2SO_4 content tested and their SS.

Comparison	Mean difference	P-value	SD
6% vs. 8%	-1.637	0.0042	Yes
6% vs. 10%	-1.633	0.0043	Yes
6% vs. 12%	-2.087	0.0005	Yes
8% vs. 10%	0.003333	>0.9999	No
8% vs. 12%	-0.45	0.7641	No

Table 5. Variation of the ethanol production parameters from yam peel as a function of hydrolysis time.

Hydrolysis time (h)	TSS (°Brix)	FA (%)	EC (% v/v)	EY (%)
0	00.00 ± 00.00	00.00 ± 00.00	0.00 ± 0.00	00.00 ± 00.00
1	15.00 ± 00.00	28.24 ± 0.79	4.69 ± 0.09	32.55 ± 0.66
2	15.17 ± 00.17	46.24 ± 1.08	6.72 ± 0.26	46.66 ± 1.77
4	16.00 ± 00.00	51.08 ± 0.54	6.65 ± 0.10	46.20 ± 0.66
6	16.50 ± 00.00	52.27 ± 0.44	7.07 ± 0.13	49.10 ± 1.29

TSS: Total Soluble Solids; FA: Final Attenuation

Comparison	Mean difference	P-value	SD
10% vs. 12%	-0.4533	0.7589	No

SS: Statistical Significance

However, an in-depth analysis of the ethanol content values of the fermented musts carried out using Tukey's test after the *one-way* ANOVA analysis (Table 4) shows that there is no statistically significant difference between the 8%, 10%, and 12% of H_2SO_4 tested (p -value > 0.05).

These results indicate that the minimum H_2SO_4 content required to achieve completely the hydrolysis of the polymers extracted from yam peels is reached from 8%. Therefore, a further increase in H_2SO_4 content does not significantly lead to a higher concentration of fermentable sugars convertible into bioethanol [14]. Consequently, the optimal H_2SO_4 content for carrying out the acid hydrolysis of the starch polymers contained in yam peels is 8%, providing an effective compromise between the bioethanol yield obtained and the concentration of acid required.

3.5. Determination of the Optimal Hydrolysis Time of Sugar Polymers Extracted from Yam Peels

To determine the optimal hydrolysis time of yam peels, a concentration of 8% H_2SO_4 and a S-L ratio of 1:5 (w/v) were used, while varying the hydrolysis time of 0-hour to 6 hours. The hydrolysates obtained were syrups, characterized with TSS values presented in Table 5. The data presented in this table reveals that with the initial time of 0 hour, the TSS of the solution was zero. However, from the 1st hour, there is a progressive increase in TSS; so the value of TSS passed from 15.00 ± 0.00 °Brix after 1 hour, up to 16.5 ± 0.00 °Brix at the end of the 6th hour of reaction. This trend suggests that as the hydrolysis reaction time increases, the catalyst acts more intensively on the depolymerization of polymer chains, particularly amylose and amylopectin derived from the peels.

However, since this increase in TSS observed over time is moderate, it means likely that the acid hydrolysis reaction gradually reaches a state of saturation, probably due to the decrease in hydrolysable components after the first hour of hydrolysis.

The values of the final attenuation (FA) of the fermented hydrolysate musts of yam peels at the end of fermentation is influenced by the hydrolysis time as recorded in Table 5. This finding confirms that the degree of availability of fermentable sugars for the yeast *Saccharomyces cerevisiae* varies significantly depending on the hydrolysis time applied to obtain the musts. Indeed, an increase in hydrolysis time improves the efficiency of ethanolic fermentation of the musts. From this study, the musts resulting from longer hydrolysis durations (more than two hours) reached FA values of at least 50% due to a greater availability of fermentable sugars in the hydrolysates for the yeast.

In Figure 4 is shown the TSS evolution during the fermentation of the different hydrolysates of yam peels. These results illustrated in Figure 4 suggest that short-duration of acid hydrolysis (less than one hour) does not generate enough fermentable sugars in the hydrolysates compared to longer hydrolysis. This incomplete hydrolysis is explained by the formation of water-soluble oligosaccharides, but these oligomers are poorly assimilated by the yeasts. Although the TSS values obtained at different hydrolysis times vary slightly, the availability of fermentable sugars for the yeast *Saccharomyces cerevisiae* differs significantly from one type of must to another.

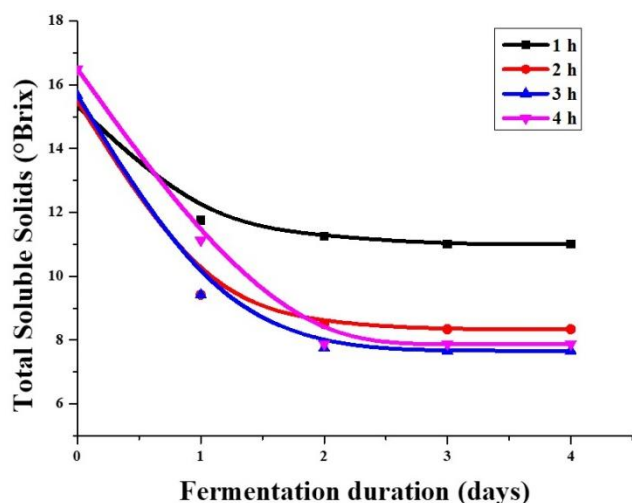


Figure 4. Evolution of the TSS of the hydrolysates as function of fermentation time.

The ethanol content (EC) produced in the fermented musts, as well as the experimental yields (EY) of bioethanol production by batch fermentation from yam peels, vary respectively from $4.69 \pm 0.09\%$ (v/v) to $7.07 \pm 0.13\%$ (v/v), and from $18.48 \pm 0.37\%$ to $27.87 \pm 0.52\%$ (Table 5). In general,

these results indicate that hydrolysis time has a notable impact on the EY of bioethanol production from yam peels and on the ethanol content of the fermented hydrolysate musts, with maximum efficiency reached at 6 hours of hydrolysis, thus producing the highest ethanol content and yield. All these findings about the hydrolysis time are in agreement with the work reported on starch hydrolysis which had evidently shown that 6-12 h was suitable for starch hydrolyzation with HCl [29].

3.6. Statistical Analysis of the Ethanol Content Produced

The statistical analysis of the ECP using Tukey's test, associated with the one-way ANOVA test (Table 6), reveals that there is no statistically significant difference between hydrolysis times of 2 h compared to 2 h, 4 h, and 6 h. Tukey's test (Table 7), as a complement to the one-way ANOVA test, is used to compare the musts using different hydrolysis durations (1 h, 2 h, 4 h, and 6 h) pairwise. It is also used to determine whether the variations between the values of EC produced are significant. The absence of statistical difference suggests that a hydrolysis duration of at least 2 h is sufficient to obtain satisfactory results, comparable to hydrolysis durations of 4 h and 6 h in terms of ECP.

Table 6. Summary of the one-way ANOVA test in the case of variation of hydrolysis time.

Parameters	Values
F	46.16
P	0.0001
SD ($P < 0.05$)	Yes
R-squared (R^2)	0.9454

In light of the various experimental results presented above, an optimal duration of 2 h is sufficient for the complete hydrolysis of the starch polymers extracted from yam peels. This result is consistent with similar studies reported in literature, such as those of [30], which emphasize the importance of optimizing hydrolysis times to maximize efficiency without compromising cost-effectiveness.

Table 7. Comparison of mean differences between the various hydrolysis times tested and their SD.

Comparison	MD:	P-value	SD
1 h vs. 2 h	-2.030	<0.0001	Yes
1 h vs. 4 h	-1.963	0.0001	Yes

Comparison	MD:	P-value	SD
1 h vs. 6 h	-2.380	<0.0001	Yes
2 h vs. 4 h	0.06667	0.9902	No
2 h vs. 6 h	-0.3500	0.4496	No
4 h vs. 6 h	-0.4167	0.3165	No

MD: Mean difference; SD: Significant difference

3.7. Effect of the DE/Water Volume Ratio on the Ethanol Production Parameters

In accordance with the results previously obtained in this study, the search for the optimum dry extract (DE)/water (W) ratio was carried out while keeping constant the acid content at 8% relative to the weight of the starchy dry extract with the hydrolysis duration of 2 hours. In Table 8 are displayed the total soluble solids (TSS), the final attenuation (F); the ethanol content (EC); and the experimental yield (EY) of ethanol production as function of DE/W ratio.

Table 8. Variation of the Parameters of Ethanol Production as a Function of the DE/W Ratio used.

DE/W (g/mL)	Cm (g/L)	TSS (°Brix)	FA (%)	EC (% v/v)	EY (%)
1:10	100	8.25 ± 0.14	30.47 ± 0.58	1.86 ± 0.08	25.89 ± 1.08
1:5	200	15.17 ± 0.17	46.24 ± 1.08	6.72 ± 0.26	46.66 ± 1.77
3:10	333	21.25 ± 0.14	36.46 ± 0.43	5.71 ± 0.19	26.42 ± 0.89
2:5	400	26.25 ± 0.14	37.14 ± 0.35	8.63 ± 0.09	29.97 ± 0.43

FA: Final attenuation; DE: dry extract; W: water TSS: Total Soluble Solids, DE: dry extract; W: water

According to DE/W ratio, the FA values of the fermented musts ranged from 8.25 ± 0.14 °Brix to 26.25 ± 0.14 °Brix. The highest TSS value was obtained with the DE/W ratio of 2:5, corresponding to 400 g/L of DE, while the lowest TSS value was observed for the DE/W ratio of 1:10 corresponding to 100 g/L of DE. These results exhibit a direct correlation between the DE concentration and the TSS obtained after hydrolysis. Probably, the quantity of polymers available in the solution to be hydrolyzed, could explain this correlation.

The ethanol contents (EC) produced with the hydrolysate musts fermented through batch mode, recorded in Table 8, show the values ranging between $1.86 \pm 0.08\%$ (v/v) and $8.63 \pm 0.09\%$ (v/v). The ratio of 2:5 proved to be the most effective for the hydrolysis of polymers derived from yam peels, because it produced the most concentrated hydrolysate (26.25 ± 0.14 °Brix) which yielded with the highest EC ($8.63 \pm 0.09\%$ (v/v)).

In Figure 5 is shown the TSS evolution during the batch fermentation of the hydrolysates as a function of the DE/W ratio used.

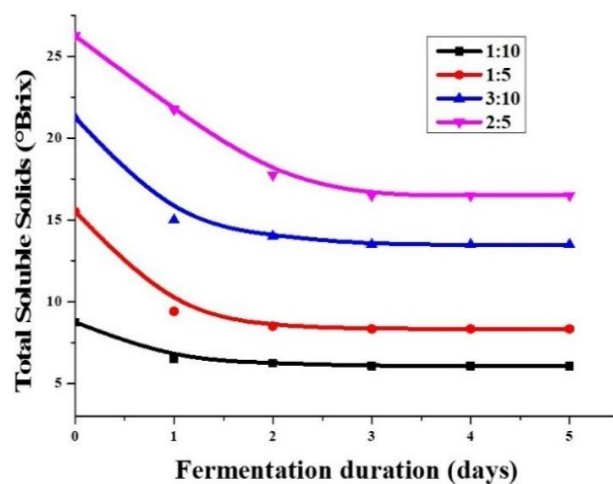


Figure 5. Evolution of the TSS of the hydrolysates as function of the DE/W ratio used.

The results reveal that the FA values ranged between $30.47 \pm 0.58\%$ and $46.24 \pm 1.08\%$. The 2:5 ratio did not yield the highest FA of the fermented hydrolysates of yam peels. In fact, it is the 1:5 ratio that produced the highest FA, which was $46.24 \pm 1.08\%$. This finding is confirmed by the EY of bio-ethanol production recorded in Table 8 such as $46.66 \pm 1.77\%$.

All of these results show that the mass concentration of 200 g/L of DE, equivalent to the 1:5 ratio, best promotes ethanolic fermentation. However, the efficiency of hydrolysis seems to be affected by an excess of DE. This could be explained by the insufficient amount of catalyst (H_2SO_4) required to break all the glycosidic bonds, leading to partial starch hydrolysis into fermentable sugars and causing the formation of oligosaccharides that are difficult for the yeast to ferment, thereby reducing fermentation yield. These observations are consistent with similar studies [30], which show that optimal S-L ratios maximize fermentation yields while avoiding practical constraints associated with excessively high biomass concentrations.

3.8. Statistical Analysis of the Results

In addition to these observations, the statistical analyses by one-way ANOVA test and Tukey's test yielded the results presented in Table 9.

Table 9. Summary of the one-way ANOVA test in the case of variation of the solid/liquid ratio used.

Parameters	Values
F	278.9
P	0.0001
SD ($P < 0.05$)	Yes
R-squared (R^2)	0.9905

SD: Significant difference

According to these results, the different DE/W ratios studied have a significant effect on fermentation yield, with a statistically outstanding difference between the means of the experimental yields. Tukey's test confirms that each DE/W ratio used is statistically distinct from the others, which validates the influence of the DE/W ratio on fermentation performance (Table 10). Thus, from this study, the DE/W ratio of 1:5 represents an optimal point for maximizing ethanolic fermentation yield under the experimental conditions, with a yield of $46.66 \pm 1.77\%$.

Table 10. Comparison of mean differences between various DE/ H_2O ratios and their SS.

Comparison	Mean difference	P-value	SD
1:10 vs. 1:5	-4.853	<0.0001	Yes
1:10 vs. 3:10	-3.843	<0.0001	Yes
1:10 vs. 2:5	-6.767	<0.0001	Yes

Comparison	Mean difference	P-value	SD
1:5 vs. 3:10	1.010	0.0130	Yes
1:5 vs. 2:5	-1.913	0.0002	Yes
3:10 vs. 2:5	-2.923	<0.0001	Yes

SS: Statistical Significance

In practice, this optimization could reduce costs and production time in an industrial setting by minimizing raw material losses while ensuring optimal bioethanol production. However, for large-scale applications, other parameters such as temperature, agitation, and the quality of the peels could also impact bioethanol production with yam peels.

3.9. Comparison of Ethanol Production Efficiency Using Four Acids as Catalysts

With the aim of optimizing bioethanol production from yam peels, four acids were tested to compare their effectiveness as hydrolysis catalysts. These included H_3PO_4 , HNO_3 , HCl , and H_2SO_4 . Each hydrolysis was carried out under the conditions such as DE/W ratio of 1:10, hydrolysis time of 2 h, and an acid content of 8% (w/w). In Figure 6 are shown the variation of TSS (°Brix) as a function of the fermentation time for the musts obtained from the four different acids used as catalysts.

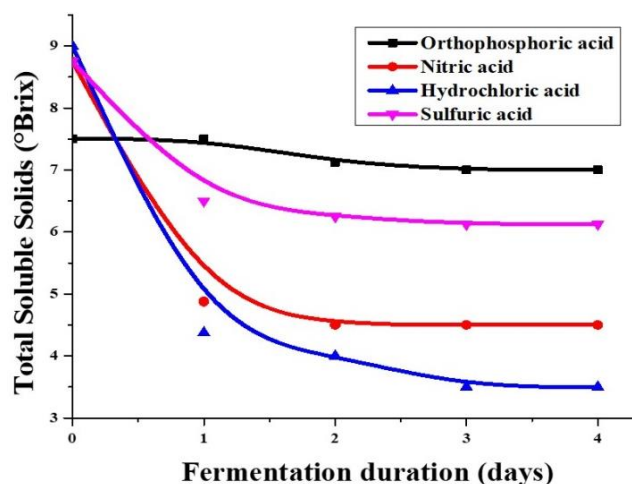


Figure 6. Evolution of the TSS of the hydrolysates during batch fermentation according to the type of acid used.

Data presented in Figure 6 confirm that HCl is more effective than the other acids studied, since fermentation of the corresponding hydrolysate provided an FA of $61.11 \pm 0.00\%$, compared with $48.57 \pm 0.00\%$; $29.99 \pm 0.33\%$; and $6.67 \pm 0.00\%$, respectively for HNO_3 , H_2SO_4 , and H_3PO_4 .

The efficiency of the four acids used as catalysts on the

ethanol production from the yam peel hydrolysates in this investigation are presented in Table 11.

Table 11. Comparison of the efficiency of the four acids on the ethanol production.

Types of acids	TSS (°Brix)	FA (%)	EC (%: v/v)	EY (%)
H ₃ PO ₄	7.5 ± 0.00	6.67 ± 0.00	0.09 ± 0.06	1.25 ± 0.81
HNO ₃	8.75 ± 0.00	48.57 ± 0.00	2.76 ± 0.08	38.35 ± 1.09
HCl	9.00 ± 0.00	61.11 ± 0.00	3.73 ± 0.12	51.74 ± 1.65
H ₂ SO ₄	8.25 ± 0.14	29.99 ± 0.33	1.86 ± 0.08	25.89 ± 1.08

TSS: Total Soluble Solids; FA: Final Attenuation; EC: Ethanol Content; EY: Ethanol Yield

The comparison of the hydrolysis results highlights that HCl produced the hydrolysate with the highest TSS (9.00 ± 0.00 °Brix), while H₃PO₄ yielded the hydrolysate with the lowest TSS (7.50 ± 0.00 °Brix). These results are consistent with the findings reported by [29], who had indicated that HCl more effectively catalyzes the breaking of glycosidic bonds in starch polymers. Other researchers also highlighted the effectiveness of HCl in producing hydrolysates richer in fermentable sugars, suitable for fermentation [31]. According to the results of this study shown in Table 11, HCl helps to achieve the highest ethanol content (EC) produced (3.72 ± 0.12% v/v), while H₃PO₄ provides the lowest EC (0.09 ± 0.06% v/v).

The EY of the ethanol production recorded in Table 11 also confirms the high performance of HCl. Indeed, this acid provided the highest EY of the ethanol production (51.74 ± 1.65%), while H₃PO₄ yielded the lowest EY of the ethanol production (1.25 ± 0.81%).

The overall analysis of the results of the current study shows that the four acids can be ranked in descending order of their starch hydrolysis efficiency as follows: hydrochloric acid > nitric acid > sulfuric acid > orthophosphoric acid. Several factors, such as the catalytic strength of each acid, the solubility of the intermediate products, and the possible presence of inhibitory effects, may account for these disparities [32, 33]. Indeed, hydrochloric acid, a strong acid characterized by low molecular steric hindrance, effectively promotes the degradation of starch polymers. It catalyzes the cleavage of glycosidic bonds optimally, increasing the release of fermentable sugars. In contrast, orthophosphoric acid, due to its low protonic capacity, slows this process and shows limited efficiency, as confirmed by the morphological analyses of hydrolyzed starches [28].

These results highlight the crucial role of the chemical properties of acids in starch hydrolysis, confirming the particular effectiveness of hydrochloric acid in optimizing the

production of fermentable sugars from yam peels.

4. Conclusion

In this study, the results show that yam peels denote a potentially rich source of starch that can be converted into bioethanol. However, it is important to work under appropriate conditions to make bioethanol production from this biomass cost-effective. Thus, the optimal conditions for hydrolysis include a sulfuric acid content of 8% (w/w), DE/W ratio of 1:5 (g/mL), and 2 h are needed to hydrolyze very well the starch polymers from yam peels. Under these aforementioned conditions, an ethanol content of 6.72 ± 0.26% (v/v) was obtained, corresponding to a yield of 264.97 ± 10.07 g of ethanol per kilogram of DE. Furthermore, the study also demonstrated that the use of hydrochloric acid gives better results compared to nitric acid, sulfuric acid, and orthophosphoric acid. Ultimately, this study paves the way for an innovative and sustainable use of agricultural waste, transforming yam peels into a valuable resource for renewable energy production. The results from this study are important for the commercial production of bioethanol from yam peels. Further studies could focus on the variation of the heating temperature as well as the use of other fermentative microorganisms, in order to improve the bioethanol production yield from this less valued agro-food residue.

Abbreviations

Ac	Ash Content
AOAC	Association of Official Analytical Chemists
DASID	Department of Agricultural Statistics, Informatics and Documentation
DE	Dry Extract
DE/W	Dry Extract/Water Volume Ratio
DMr	Dry Matter Ratio
EC	Ethanol Content
EY	Experimental Yield
FA	Final Attenuation
OMc	Organic Matter Content
SEM	Standard Error of the Mean
S-L	Solid-Liquid
SS	Statistical Significance
TSS	Total Soluble Solids
WVc	Water and Volatile Matter Content
Ac	Ash Content

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Author Contributions

Kosi Mawuena Novidzro: Conceptualization, Data curation, Formal Analysis, Project administration, Resources, Supervision, Validation, Writing – review & editing

Gnimdou Issanga Abli: Data curation, Formal Analysis, Investigation, Methodology, Software, Writing – original draft

Sassou Megnassan: Formal Analysis, Investigation, Methodology, Validation

Kossi Honore Koumaglo: Validation, Visualization

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Conflicts of Interest

The authors declare no conflicts of interest.

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