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Hydrolysis of Solid Waste From Bioethanol Plants into Glucose Using Hydrochloric Acid Catalyst

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Abstract
The large amount of solid waste from bioethanol plants is regrettable if it is only used as animal feed. The solid waste has a relatively high carbohydrate content of 36.85%, which has the potential to be hydrolysed to glucose. Hydrolysis with water is slow, so a catalyst is needed to speed up the reaction. HCl hydrolysis is commonly used to convert carbohydrates to glucose. The hydrolysis process in this study involved heating at 95°C with a stirring speed of 300 rpm. The variables tested were hydrolysis time (90, 120, 150, 180, 210 minutes) and HCl concentration (1.5, 2, 2.5, 3, 3.5N). The results showed that hydrolysis time and HCl concentration had a direct effect on glucose yield. The longer the hydrolysis time and the higher the concentration used, the higher the glucose yield. The highest glucose content obtained was 23.12%, with a yield of 70.6%, obtained under the conditions of 210 minutes hydrolysis time and 3.5 N HCl concentration.
Keywords: *Carbohydrate, Glucose, HCl, Hydrolysis, Yield*

Abstrak
Limbah padat dari pabrik bioetanol mengandung kandungan karbohidrat yang relatif tinggi yaitu 36,85%, yang berpotensi untuk dihidrolisis untuk menghasilkan glukosa. Hidrolisis dengan air terjadi secara perlahan, sehingga membutuhkan katalis untuk mempercepat reaksi. Hidrolisis HCl umumnya digunakan untuk mengubah karbohidrat menjadi glukosa. Proses hidrolisis pada penelitian ini melibatkan pemanasan pada suhu 95°C dengan kecepatan pengadukan 300 rpm. Variabel yang diuji adalah waktu hidrolisis (90, 120, 150, 180, 210 menit) dan konsentrasi HCl (1,5, 2, 2,5, 3, 3,5 N). Hasil penelitian menunjukkan bahwa waktu hidrolisis dan konsentrasi HCl berpengaruh langsung terhadap yield glukosa. Semakin lama waktu hidrolisis dan semakin tinggi konsentrasi yang digunakan, maka semakin besar pula hasil glukosa yang diperoleh. Kadar glukosa tertinggi yang diperoleh adalah 23,12%, dengan rendemen 70,6%, dicapai pada kondisi waktu hidrolisis 210 menit dan konsentrasi HCl 3,5N.
Kata Kunci: *Glukosa, HCl, Hidrolisis, Karbohidrat, Yield*

1. Introduction

The solid waste from bioethanol plants is the byproduct generated after the fermentation process of molasses into bioethanol. This solid waste, along with the wastewater from fermentation, exits towards the primary settling in the form of slurry. In each fermentation process of molasses into bioethanol, PT Energi Agro Nusantara produces abundant tons of solid waste [1]. The solid yeast waste from breweries is also a byproduct of the fermentation process, with a chemical composition of 25-35% non-cellulosic carbohydrates, 17-25% cellulose, 15-24% protein, and the remainder consisting of lignin, lipids, and ash [2]. The relatively high carbohydrate content makes it highly feasible to reprocess the solid waste from bioethanol plants into glucose. This is similar to Pertiwi's 2016 research on converting the solid waste from brem liquid (an alcoholic beverage) into liquid sugar. The highest yield of reducing sugar from the hydrolysis process was obtained under conditions of 30 minutes and a ratio of solid brem waste to HCl of 1:4, resulting in 33.89 g/ml. [3].

The commonly used method to convert carbohydrates from bioethanol plant solid waste into glucose is hydrolysis. The hydrolysis process involves breaking down compounds by reacting them with water, with the goal of decomposing them into simpler substances. The word 'hydrolysis' comes from Greek, with 'Hydro' meaning 'water,' and 'Lysis' meaning 'to release,' so the term hydrolysis implies the breaking down of a substance with water. Simply put, starch hydrolysis is a process where starch molecules are broken down into simpler components such as glucose. The reaction for the hydrolysis of starch into glucose is as follows:

$$(C_6H_{10}O_5)n \xrightarrow[H^+]{nH_2O} nC_{12}H_{22}O_{11}$$

starch Maltose

$$(C_{12}H_{22}O_{11})n \xrightarrow[H^+]{nH_2O} nC6H12O6$$

Maltose Glucose

[4]

Starch is a type of polysaccharide carbohydrate, composed of anhydro monosaccharide polymers with the general formula $(C_6H_{10}O_5)n$. The main components of starch are amylose and amylopectin. Amylose consists of glucose units linked by 1-4 glycosidic bonds, while amylopectin is a polysaccharide composed of 1-4α glycosidic bonds with 1-6α glycosidic branches. During the hydrolysis reaction of starch with water, the water attacks the starch at the 1-4α glycosidic bonds, breaking it down into shorter chains. The result can be dextrin, syrup, or glucose, depending on the degree of polysaccharide chain breakdown in the starch. If the ratio of suspension and time is correct, the dextrin formed will be further hydrolyzed into glucose. The hydrolysis process with water occurs slowly, so a catalyst is needed to speed up the reaction rate [5].

The catalysts commonly used in the hydrolysis process of converting starch into glucose are either enzymes or chemical agents such as acids [6]. Acid hydrolysis is more commonly used because it does not require any pretreatment [7]. Acid hydrolysis also requires a shorter time compared to enzymes and is more cost-effective [8]. Common acid catalysts used for the hydrolysis of starch into glucose include H_2SO_4, HCl, and HNO_3. Research by Hartiati in 2018 on the hydrolysis of carbohydrate content in taro tubers with various acid HNO_3, HCl, and H_2SO_4 showed that the highest reducing sugar content among these three acids, under conditions of hydrolysis temperature at 100°C and acid concentration at 7%, was achieved using HCl, which produced the highest reducing sugar value of 3.06% [9]. Research conducted by Rizal in 2016 entitled “Hydrolysis of cellulose from cocoa husk pod material” which states that the hydrolysis process with hydrochloric acid has the advantage that the salt formed after neutralizing the results is harmless salt (kitchen salt)[10].

Factors influencing the hydrolysis process include: particle size of the material, material ratio, hydrolysis time, acid concentration, and stirring [11]. Acid concentration affects the glucose yield. Inayati's 2014 study on the hydrolysis of potato starch with HCl at various concentrations (0.5, 1, 1.5, 2 N) showed that with each increase in acid concentration, the glucose content increased. The highest glucose yield was obtained at an HCl concentration of 2 N, reaching 2600 mg [12]. Hydrolysis time also affects the glucose yield. Safitri's 2022 study on the production of liquid glucose from yellow sweet potatoes using acid hydrolysis, with time variables of 60, 90, and 120 minutes, showed that as the hydrolysis time increased, the glucose content also increased. The best result was achieved with a hydrolysis time of 120 minutes, yielding a glucose concentration of 5.81% [13].

2. Material and Methods

In this study, the hydrolysis of carbohydrates into glucose was performed using acid catalysts. The hydrolysis process was carried out with heating and stirring.

A. Materials And Equipment

The main raw material, which is the solid waste from the bioethanol plant, was obtained from PT. Energi Agro Nusantara. Hydrochloric acid was purchased from a chemical store in Surabaya. The hydrolysis setup includes an overhead stirrer, clamps, stand, thermometer, heating mantle, three-neck flask, and condenser (as shown in **Figure 1**).

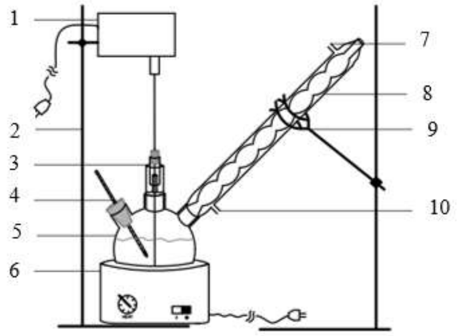

Figure 1. Hydrolysis apparatus circuit

B. Preparation Of Raw Materials

1. Hydrochloric Acid Solution Preparation
   Hydrochloric acid (HCl) 98% was diluted with distilled water to achieve the concentrations used in this study: 1.5 N, 2 N, 2.5 N, 3 N, and 3.5 N.
2. Solid Yeast Waste Preparation
   The solid waste from PT. ENERO, which was initially in slurry form, was dried using sunlight (32-36°C) until a constant weight was achieved. The dried material was then size-reduced and sieved through a 100 mesh screen to obtain powdered solid waste. The powdered solid waste was tested for carbohydrate, protein, cellulose, ash, and fat content.

C. Hydrolysis

Assemble the hydrolysis apparatus as shown in **Figure 1**. Weigh 50 grams of powdered solid waste from the bioethanol plant and transfer it into a three-neck flask. Then, add 500 ml of hydrochloric acid solution of the desired concentrations into the flask containing the powdered waste. Turn on the stirrer and set it to a stirring speed of 300 rpm. Set the heating mantle to maintain a temperature of 95°C and proceed with the hydrolysis for the specified duration. Once the hydrolysis is complete, cool the glucose solution and store it in a storage bottle. Measure the density of the obtained glucose solution and test the glucose content using the Luff-Schoorl method and FTIR analysis.

3. Results and Discussion

The solid waste from the bioethanol plant at PT. Energi Agro Nusantara, which exits from the lamella clarifier, is in slurry form. The solid waste is then dried to produce a powder. Subsequently, a material content analysis was conducted at the Research and Industrial Consultation Center (BPKI) in Surabaya, and the results are shown in **Table 1**.

Table 1. Composition of Bioethanol Plant Solid Waste

Component	Value (%)
Carbohydrate	36,85
Cellulose	18,62
Proteins	5,02
Lipids	3,01
Ash	1,26

The analysis results show a significant carbohydrate content of 36.85%. This carbohydrate content can be utilized to produce glucose. Based on the research on the hydrolysis of solid waste from bioethanol plants conducted at a heating temperature of 95°C and stirring speed of 300 rpm, with various HCl concentrations and hydrolysis times, the glucose content was measured using the Luff-Schoorl method, tested at the Balai Penelitian Dan Konsultasi Industri, Surabaya, as shown in **Table 2**.

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Table 2. The results of the glucose content tests and glucose yield calculations from the hydrolysis of solid waste from bioethanol plants into glucose, across various HCl concentrations and hydrolysis times

HCl Concentration (N)	Hydrolysis time (min)	Glucose level (%)	Yield glucose (%)
1,5	90	14.82	45.25%
	120	16.32	49.83%
	150	16.52	50.44%
	180	17.02	51.97%
	210	17.65	53.89%
2	90	16.54	50.50%
	120	17.85	54.50%
	150	19.02	58.08%
	180	19.72	60.21%
	210	20.01	61.10%
2,5	90	17.8	54.35%
	120	18.05	55.11%
	150	19.88	60.70%
	180	20.15	61.53%
	210	20.86	63.70%
3	90	18.9	57.71%
	120	20.1	61.37%
	150	21.15	64.58%
	180	22.03	67.27%
	210	22.11	67.51%
3,5	90	20.05	61.22%
	120	20.24	61.80%
	150	21.61	65.99%
	180	23.01	70.26%
	210	23.12	70.60%

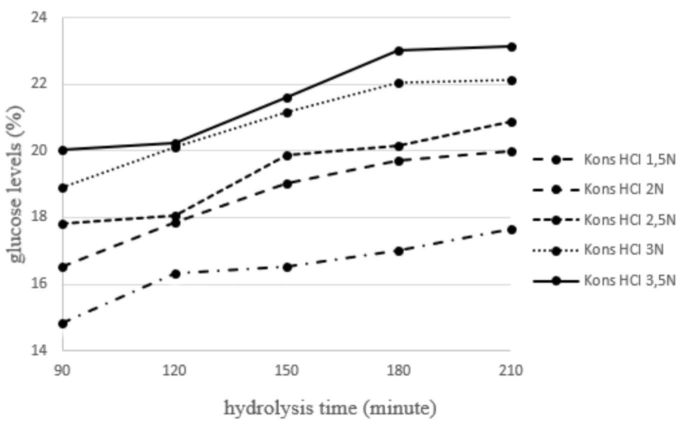

Figure 2. Relationship Between Glucose Content, HCl Concentration, and Hydrolysis Time

Based on **Figure 2**, it can be observed that the duration of hydrolysis affects the glucose yield. During the hydrolysis time of 90-180 minutes, the glucose content increases progressively. However, between 180-210 minutes, the increase in glucose content becomes less significant or nearly constant. This is due to the decreasing amount of starch, the reactant, as the hydrolysis time extends. The reduction in starch content leads to a less significant increase in glucose yield. Overall, the increase in glucose content is influenced by the duration of hydrolysis, as a longer hydrolysis time results in more frequent collisions between reactants, leading to a higher reaction rate and thus an increased glucose yield.

The concentration of HCl also affects the glucose content produced. An increase in HCl concentration from 1.5 to 2 N results in a substantial increase in glucose yield. Similarly, increasing the concentration from 2 to 3 N also raises glucose yield, although not as significantly as the 1.5 to 2 N range. At concentrations from 3 to 3.5 N, the increase in glucose yield is minimal. HCl acts as a catalyst, breaking the 1-4 α-glycosidic bonds in starch. A higher HCl concentration means a higher number of available H+

ions to break these bonds. Therefore, increasing the HCl concentration results in more H+ ions, leading to more bond cleavage and consequently a higher glucose yield. This is consistent with Yustina's 2018 study, which found that a higher acid catalyst concentration accelerates the hydrolysis reaction, resulting in increased glucose production [14].

Table 2 also shows the calculated glucose yield results. As the concentration of HCl increases, the glucose yield also increases. Similarly, a longer hydrolysis time results in a higher glucose yield. These results are illustrated in detail in **Figure 3** below.

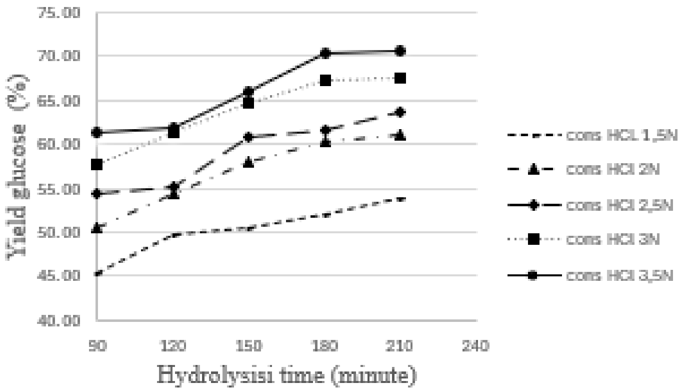

Figure 3. Relationship Between Glucose Yield, HCl Concentration, and Hydrolysis Time

Figure 3 shows that both hydrolysis time and HCl concentration affect glucose yield. At hydrolysis times ranging from 90 to 210 minutes, the calculated glucose yield increases. Longer hydrolysis times result in higher glucose yields because more carbohydrates are hydrolyzed into glucose. For HCl concentrations between 1.5 and 3.5 N, the yield increases with higher acid concentrations. The highest glucose yield of 70.6% was achieved with 3.5 N HCl and a hydrolysis time of 210 minutes. Conversely, the lowest glucose yield of 45.25% was observed with 1.5 N HCl and a hydrolysis time of 90 minutes.

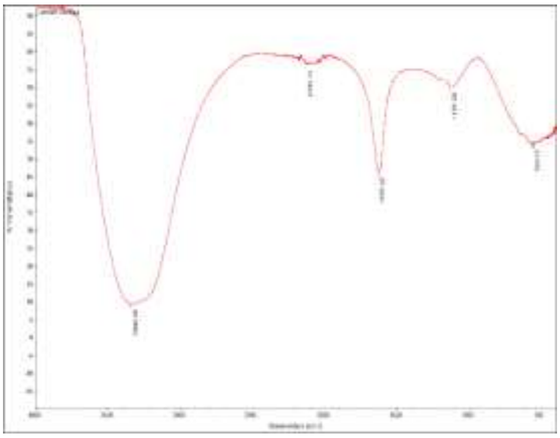

Figure 4. FTIR Analysis Results for Glucose Hydrolysate Solution

The FTIR analysis results from **Figure 4** shows several absorption peaks at wavelengths of 3342.26 cm^{-1}, 2135.14 cm^{-1}, 1635.42 cm^{-1}, 1135.29 cm^{-1}, and 563.75 cm^{-1}. The highest peak is observed at a wavelength of 3342.26 cm^{-1}, indicating the presence of hydroxyl groups (–OH), which are characteristic of alcohol compounds. The peak at 1635.42 cm^{-1} represents the C=C stretch, which is associated with alkenes. The wavelength of 1135.29 cm^{-1} indicates C-O bonds, which can be found in alcohols, ethers, carboxylic acids, and esters [15].

4. Conclusion

The carbohydrate content in the solid waste from the bioethanol plant is 36.85%. The highest glucose content was obtained with an HCl concentration of 3.5 N and a hydrolysis time of 210 minutes, reaching 23.12% glucose with a yield of 70.6%. Variations in HCl concentration and hydrolysis time in the production of glucose from solid waste of the bioethanol plant can enhance the glucose content achieved.

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