Produksi Gula Pereduksi dari Batang Kelapa Sawit melalui Proses Hidrolisis Asam dengan Variasi Konsentrasi Asam Klorida (HCl)

Production of Reducing Sugars from Oil Palm Stems through Acid Hydrolysis Process with Variations in Hydrochloric Acid (HCl) Concentrations

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ABSTRAK

Batang kelapa sawit termasuk salah satu limbah perkebunan yang melimpah dan mengandung selulosa serta hemiselulosa yang dapat dikonversi menjadi gula pereduksi melalui proses hidrolisis asam. Penelitian ini bertujuan untuk mengevaluasi pengaruh konsentrasi asam klorida (HCl) terhadap produksi gula pereduksi dari hidrolisat batang kelapa sawit. Penelitian menggunakan tiga perlakuan konsentrasi HCl, yaitu 1 M, 2 M, dan 3 M, masing-masing dengan tiga kali ulangan. Sampel terlebih dahulu mengalami pretreatment menggunakan larutan NaOH 1 M, kemudian dihidrolisis pada suhu 80–90°C selama 100 menit. Parameter yang diamati meliputi total padatan terlarut (Brix) yang dianalisis menggunakan refraktometer dan kadar gula pereduksi yang dianalisis menggunakan metode Nelson-Somogyi. Hasil penelitian menunjukkan bahwa peningkatan konsentrasi HCl meningkatkan produksi gula. Nilai rata-rata °Brix meningkat dari 6% pada perlakuan 1 M menjadi 13% pada perlakuan 3 M, sedangkan kadar gula pereduksi meningkat dari 366,25 ppm menjadi 508,07 ppm. Dengan demikian, batang kelapa sawit memiliki potensi sebagai bahan baku bioetanol, dan perlakuan HCl 3 M merupakan kondisi hidrolisis yang paling efektif dalam penelitian ini.

Kata kunci: Batang Kelapa Sawit, Hidrolisis Asam, Asam Klorida, Gula Pereduksi, Bioetanol generasi kedua

ABSTRACT

Oil palm trunks are one of the most abundant types of plantation waste and contain cellulose and hemicellulose that can be converted into reducing sugars through an acid hydrolysis process. This study aimed to evaluate the effect of hydrochloric acid (HCl) concentration on reducing sugar production from oil palm trunk hydrolysate. The experiment employed three HCl concentration treatments, namely 1 M, 2 M, and 3 M, each with three replications. Before hydrolysis, the samples were pretreated using 1 M NaOH solution and subsequently hydrolyzed at 80–90°C for 100 minutes. The parameters observed included total dissolved solids (Brix), measured using a refractometer, and reducing sugar content, determined using the Nelson-Somogyi method. The results showed that increasing HCl concentration enhanced sugar production. The average Brix value increased from 6% at 1 M HCl to 13% at 3 M HCl, while the reducing sugar content increased from 366.25 ppm to 508.07 ppm. Therefore, oil palm trunk has considerable potential as a feedstock for bioethanol production, and the 3 M HCl treatment was found to be the most effective hydrolysis condition in this study.

Keywords: *oil palm trunk, acid hydrolysis, hydrochloric acid, reducing sugar, second-generation bioethanol*

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INTRODUCTION

Energy demand in Indonesia continues to increase in line with population growth, industrial development, and increasing economic activity. Until now, the fulfillment of national energy needs still depends on fossil fuels derived from non-renewable natural resources. High dependence on fossil energy has the potential to lead to reduced petroleum reserves, increased dependence on energy imports, and environmental impacts in the form of greenhouse gas emissions. Therefore, the development of renewable energy sources, such as bioethanol, is one of the strategic efforts in supporting national energy security (Al Hakim, 2020; Desti, 2022).

As one of the renewable energy sources, bioethanol has good potential in reducing dependence on fossil energy while reducing greenhouse gas emissions. The production of second-generation bioethanol makes extensive use of lignocellulose biomass because it is abundant, renewable, and does not interfere with the availability of food. This biomass is composed of cellulose, hemicellulose, and lignin components, with cellulose and hemicellulose that can be converted into fermented sugars through the hydrolysis process, which is then used in the fermentation process for bioethanol production. (Ocreto et al., 2021).

Oil palm stems are plantation waste that is available in abundant quantities from plant replanting activities. This biomass contains lignocellulose and sugar that have the potential to be converted into second-generation bioethanol so that it can increase the added value of waste while supporting the development of sustainable renewable energy (Billateh & Cheirsilp, 2024).

Acid hydrolysis is a widely used method because the process is relatively fast and capable of producing high reducing sugars. In bioethanol production, the hydrolysis process plays an important role because it aims to decompose polysaccharides into reducing sugars that can be fermented by microorganisms. The amount of simple sugar produced at this stage greatly determines the success of the subsequent fermentation process. Therefore, optimization of hydrolysis parameters needs to be carried out to obtain higher reducing sugar levels and increase the efficiency of bioethanol production (Thahir et al., 2024; Ma'mun et al., 2024)

HCl is one of the effective catalysts in the hydrolysis of lignocellulose biomass because it is able to convert cellulose and hemicellulose into fermentable sugars through the breaking of β-glycosidic bonds. In addition, the use of HCl can increase the porosity of biomass and increase the accessibility of the substrate to hydrolysis reactions so that the sugar release process takes place more optimally. Research shows that hydrolysis using HCl is able to produce high reducing sugars with relatively low by-product formation (Gómora-Hernández et al., 2020).

Despite its great potential, the lignocellulose structure in oil palm stalks is relatively complex due to the presence of lignin that protects cellulose and hemicellulose from degradation. Therefore, a pretreatment stage is needed to reduce the lignin content and increase the accessibility of cellulose to the hydrolysis process. Pretreatment using alkaline solutions such as sodium hydroxide (NaOH) has been widely used because it is effective in partially dissolving lignin and increasing the porosity of biomass (Tareen et al., 2020).

Oil palm stalks are one of the lignocellulose biomasses that has the potential to be a raw material for second-generation bioethanol because it contains a fairly high amount of cellulose, hemicellulose, and lignin. Composition analysis showed that oil palm stalks contained 38.67% cellulose, 30.22% hemicellulose, and 23.76% lignin based on the dry weight of biomass. The cellulose and hemicellulose content can be hydrolyzed into simple sugars, which are then fermented into bioethanol (Tareen et al., 2020)

In addition, concentrated acid hydrolysis also has the potential to produce by-products in the form of furfural from pentose decomposition and hydroxymethylfurfural (HMF) from hexose decomposition which can inhibit the subsequent fermentation process (Świątek et al., 2020). Therefore, optimizing acid concentration is an important key to maximizing sugar yield while minimizing the formation of these inhibitors. Prior to hydrolysis, alkaline pretreatment using NaOH was performed to degrade lignin and improve the accessibility of cellulose and hemicellulose to acid catalysts (Tareen et al., 2020).

Although various studies have examined acid hydrolysis in lignocellulose biomass, specific studies on the effect of variations in HCl concentration on the production of reducing sugars from oil palm stems are still very limited. Most previous studies on oil palm stalks used sulfuric acid (H_2SO_4) as a catalyst, while the use of HCl as an alternative to mineral acids that is more manageable has not been studied systematically. In addition, there are no adequate quantitative data on the relationship between HCl concentration (1–3 M) and oil palm stem hydrolysate quality parameters, including total dissolved solids (°Brix) and reducing sugar levels measured using the standardized Nelson-Somogyi method. Therefore, this study aims to examine the effect of variations in HCl concentration (1 M, 2 M, and 3 M) on the total dissolved solids and reducing sugar content of oil palm stem hydrolysate that has undergone NaOH pretreatment, in order to identify the most effective hydrolysis conditions to support the production of second-generation bioethanol based on oil palm plantation waste (Giyanto et al., 2023; Mawarda Rilek et al., 2017).

MATERIALS AND METHODS

Tools and Materials

The tools used include an oven, a 100 mesh sieve, an analytical balance, a beaker cup, a hot plate, a pH meter, a magnetic stirrer, a refractometer, and a UV-Vis spectrophotometer (UV-1800, Shimadzu Corporation, Japan).

The materials used are palm stalks; Sodium Hydroxide (NaOH) with analytical grade purity (≥98%) was used in this study. The NaOH in pellet form was purchased from Merck KGaA (Germany) solution 1 M, hydrochloric acid (HCl) 1 M, 2 M, 3 M; distilled water; Standard sugar solution; Nelson-Somogyi Reagent, Arsenomolybdate 25 ml.

Preparation of raw materials

The inner part of the oil palm trunk is used as raw material. The oil palm trunk is then shredded and pressed to reduce its moisture content, after which it is dried in the sun. The dried material is then ground and sieved using a 100-mesh sieve to produce oil palm trunk powder.

Pretreatment

A total of 100 grams of oil palm stem powder is put into a glass beaker, and 1 liter of 1 M NaOH solution is added. The mixture is heated to 90°C with stirring at 200 rpm for 30 minutes, then the sample is washed using distilled water to a neutral pH. The next stage is to dry the sample using an oven at 70°C for 2 hours. (Giyanto et al., 2023).

Hydrolysis

The sample of 4 g of pretreatment results was placed in a 150 mL beaker glass, then mixed with 100 mL of HCl solution with a concentration of 1 M, 2 M, or 3 M according to the respective treatment conditions. Hydrolysis takes place at a temperature of 80°C for 100 minutes with the help of a magnetic stirrer operated at a speed of 200 rpm. After the hydrolysis stage ends, the hydrolysate is left to reach room temperature before undergoing dissolved solids testing and reducing sugar content measurements. (Mawarda Rilek et al., 2017).

Neutralization

The hydrolysis sample is neutralized before reducing sugar analysis is carried out.

Once the hydrolysis process is complete, the beaker is removed from the hot plate, and the solution is left on the laboratory bench until its temperature naturally decreases to room temperature (±25–30°C). Then, NaOH solution is gradually added while stirring using a stirrer until the pH reaches 6.5–7.0, which is measured using a pH meter. Once a neutral pH is achieved, the sample is centrifuged or filtered to separate the solid residue and impurities, and the filtrate is used for reducing sugar analysis (Świątek et al., 2020).

The neutralized hydrolysate was filtered before further analyses. Total soluble solids (°Brix) were measured using a refractometer and interpreted as an indicator of total dissolved solids (TDS). Since refractometric measurements are based on refractive index, dissolved salts formed during neutralization may also contribute to the measured values (Nasiri et al., 2018; Jaywant et al., 2022).

Reducing Sugar Levels Analysis

The analysis of reducing sugars was carried out using the Nelson–Somogyi method. A total of 1 mL of sample was mixed with 1 mL of Nelson reagent and heated for 20 minutes. After cooling, 1 mL of arsenomolybdate reagent and 7 mL of distilled water were added to obtain a final volume of 10 mL. Prior to analysis, the sample was subjected to a five-fold dilution (dilution factor = 5) to ensure the absorbance values fall within the linear range of the calibration curve. The absorbance of the solution was measured using a UV–Vis spectrophotometer at 540 nm. The concentration of reducing sugars was determined using a glucose standard curve prepared at concentrations of 0, 15, and 70 (Aksari et al., 2024)

$$\text{Reducing Sugar Levels} = \frac{(y-c)}{m} \times FP$$

where:

y = Sample absorbance value

c = Intercept value

m = Slope value

FP = Dilution factor

RESULTS AND DISCUSSION

Total Dissolved Solids (Brix) After Hydrolysis

Table 1. Dissolved solids (Brix) data after hydrolysis

Treatment	Repetition (%)			Average (%)
	I	II	III	
1 M	5	7	6.5	6.17
2 M	10	8.4	8.3	8.90
3 M	13	12	14	13

Based on Table 1, it can be seen that the increase in the concentration of hydrochloric acid (HCl) affects the amount of dissolved sugar (°Brix) produced from the hydrolysis process of palm stalks. Control treatment using distilled water yielded a dissolved solids value of 0%, which indicates that in the absence of an acid catalyst, the hydrolysis process did not take place effectively. In the treatment of HCl 1 M, an average dissolved solids value of 6.17% was obtained, then increased to 8.90% at the concentration of 2 M HCl, and reached the highest value of 13% at the concentration of 3 M HCl. The increase in dissolved solids content was influenced by the addition of acid during hydrolysis. The process of heating and adding acid can damage the cell structure and membrane in the sample so that previously bound dissolved sugars can escape the cell.

The ANOVA results on the dissolved solids parameter showed a calculated F value of 35.567, which is significant. This value shows that variations in HCl concentrations (1 M, 2 M, and 3 M) have a very pronounced effect on the level of hydrolysed solids in the hydrolysate of palm stalks. Thus, the difference in the average Brix between treatments is not due to a coincidence factor or random error, but is actually influenced by the difference in acid concentration used during the hydrolysis process.

The increase in dissolved solids levels in this study showed that higher concentrations of HCl increased the effectiveness of hydrolysis of lignocellulose of oil palm stalks; the breaking of β-1,4-glycosidic bonds in cellulose and hemicellulose was catalyzed by the H^+ ions of HCl, as stated by Zhou et al. (2021). This process produces simple sugars that are soluble in the reaction medium. In addition, heating at a temperature of 80°C accelerates the penetration of acids into the biomass structure. This condition causes the amount of sugar dissolved in the hydrolysate to increase as the concentration of acid used increases. In addition, the heating process during hydrolysis also helps to damage the cell structure of the material so that the previously bound sugars can dissolve into the solution.

The increase in °Brix values in this study indicates an increase in the amount of dissolved solids during the hydrolysis process. These results are consistent with those of (Ayuni & Hastini, 2020), who reported that an increase in HCl concentration can enhance the formation of simple sugars through the cleavage of glycosidic bonds in cellulose and hemicellulose, thereby increasing the conversion of lignocellulosic biomass into fermentable sugars. However, the °Brix value cannot be interpreted directly as the glucose or reducing sugar content because it is measured based on the refractive index of the solution, which represents the total dissolved solids in the hydrolysate (Jaywant et al., 2022). In addition to sugars, other components such as neutralization salts, organic acids, and lignin degradation products also contribute to the °Brix value. Therefore, the °Brix value tends to be higher than the reducing sugar content measured by the DNS method, since DNS specifically measures only reducing sugars, whereas °Brix encompasses all dissolved components in the solution (Rysha & Mulaj, 2026).

The increase in reducing sugar levels in this study showed that the hydrolysis process was able to convert the lignocellulose component of oil palm stems into simple sugars more effectively at higher concentrations of HCl (Purnawan et al., 2021).

Reducing Sugar Levels of Oil Palm Stems after the Hydrolysis Process

Table 2. Reducing Sugar Data After Hydrolysis

HCl Concentration (M)	Reducing Sugar (ppm)	Yield (%)
1	366.25	0.92
2	452.22	1.13
3	508.07	1.27

Table 2 shows a positive correlation between the concentration of HCl used and the resulting reducing sugar content, with the reducing sugar content increasing from 366.25 ppm in the 1 M HCl treatment to 508.07 ppm in the 3 M HCl treatment. Based on the ratio of the total mass of reducing sugar produced to the initial dry mass of the raw material, the percentage yield of reducing sugar was 0.92% for the 1 M treatment, 1.13% for the 2 M treatment, and 1.27% for the 3 M treatment. However, these relatively low yields are thought to be due to the presence of silica particles in oil palm tissue, which act as a natural defense mechanism and can reduce sugar monomer yields during the hydrolysis process, as well as the partial degradation of reducing sugars into furfural and HMF under highly acidic conditions, given that increased sugar concentrations in the system also increase HMF formation, at a higher rate with greater hydrolysis severity (Bishop et al., 2025; Santanaraj et al., 2017).

However, the reducing sugar values obtained were significantly lower than the Brix values measured in the same samples. This discrepancy is not due to analytical error but is a logical consequence of the fundamental differences between the two methods. The Brix scale measures the refractive index of a solution calibrated against a sucrose-equivalent scale;

thus, the reading represents the total accumulation of all dissolved substances in the solution, not sugar specifically. This is confirmed by Li et al., (2021), who state that total dissolved solids (Brix) is a quality parameter indicating the overall proportion of dissolved substances in a solution, which is a combination of sugars, organic acids, and other minor components such as phenolics, amino acids, soluble pectin, ascorbic acid, and minerals rather than the contribution of sugars alone.

The results of the analysis showed that the F value was calculated at 12,021 with a significant value. Since the significance value is less than 0.05, it can be concluded that the variation in HCl concentration (1 M, 2 M, and 3 M) exerts a very pronounced influence on the level of dissolved solids of palm stalks.

The increase in reducing sugar levels observed in this study indicates the effective degradation of hemicellulose and cellulose into simple sugar monomers. These findings are consistent with the results of a study by (Świątek et al., 2020), who reported that acid hydrolysis promotes the decomposition of lignocellulosic components, resulting in the formation of soluble sugars such as glucose, xylose, and mannose. Similarly, (Zhou et al., 2021) explained that hydrochloric acid acts as a catalyst by providing H^+ ions that facilitate the cleavage of β-1,4-glycosidic bonds in cellulose and hemicellulose, thereby increasing the release of fermentable sugars. By comparison, this study also shows that increasing the HCl concentration enhances hydrolysis efficiency, as evidenced by higher reducing sugar content in the hydrolysate. These results confirm that acid concentration plays a crucial role in accelerating the degradation of lignocellulose and maximizing sugar yield during hydrolysis.

Increased acid concentrations increase the depolymerization process of cellulose and hemicellulose as more H^+ ions are available to break the glycosidic bonds that make up polysaccharides. This condition causes the formation of simple sugars, such as glucose and xylose, to increase so that the reduced sugar levels obtained are higher (Zhou et al., 2021).

CONCLUSION

Based on the results of the study, it can be concluded that the variation in hydrochloric acid (HCl) concentration has an effect on the dissolved sugar content and the reducing sugar content resulting from the hydrolysis of oil palm stalks. Increasing the concentration of HCl from 1 M to 3 M increased the total value of dissolved solids (°Brix) from 6% to 13% and increased the reducing sugar level from about 366.25 ppm to 508.07 ppm, and the yield of reducing sugar was from 0.92% to 1.27%. The results of the analysis showed that higher concentrations of HCl were able to accelerate the breakdown of polysaccharide bonds in cellulose and hemicellulose, resulting in greater amounts of simple sugars. The concentration of 3 M HCl is the optimal condition in the tested treatment range and has the potential to be applied as a fermentable sugar production stage for second-generation bioethanol based on palm oil stalks.

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