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Effect of Hydrolysis Temperature and Acid Solution Concentration on Hydrolysis Of Hyacinth

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Abstract. The purpose of this study was to convert cellulose into glucose from water hyacinth plantsor Eichornia crassipes through acid hydrolysis with variations in temperature, acid concentration, and materials (parts of water hyacinth: stems, leaves, and mixtures stems-leaves) to obtain the best acid hydrolysis conditions based on glucose values obtained. Thehydrolysis temperatures used were 90°C, 100°C, and 110°C. The required hydrolysis timeis 90 minutes. The type of acid used is H2SO4 with concentrations of 2%, 4%, 6%, 8%,10%, and 12%. The materials used are water hyacinth stems, leaves, and mixtures. Based on the results of the study obtained glucose with the highest concentration of 0.1788 g/mLas much as 9 mL from hydrolysis with an acid concentration of 12% at a temperature of100°C and the material used was stems with the acquisition of % yield of glucose to drywater hyacinth mass of 44, 51% and % yield of glucose to the mass of water hyacinthby 5.34%.

INTRODUCTION

One of the waters in Indonesia that is filled with water hyacinth plants is the Cirata Reservoir. Cirata Reservoir is located in West Bandung Regency, West Java. Nearly 70% of the Cirata Reservoir is filled with water hyacinth plants. Floating net cage farmers in the waters of the Cirata Reservoir complained about the widespread presence of water hyacinth weeds. Covering the water surface with water hyacinth plants results in reduced oxygen and exposure to sunlight. Thus, the fish in the waters of the Cirata Reservoir are deprived of oxygen. However, in addition to causing negative impacts, water hyacinth plants have benefits in preventing the accumulation of heavy metals, organic fertilizers, fungal growth media and have great potential in the manufacture of biomass such as bioethanol. Water hyacinth contains cellulose 64.51% hemicellulose 15.61% lignin 7.69%. The cellulose contained is broken down into glucose through a hydrolysis process [1]. The glucose will be fermented into bioethanol.

Before hydrolysis of water hyacinth was carried out, pretreatment was carried out by adding 0.5 M NaOH to water hyacinth which had been equalized in size to 1 cm. The process is a delignification that aims to remove the lignin content. This lignin content needs to be removed because it can hinder the hydrolysis process in converting cellulose into glucose [2]. After the delignification stage, the hydrolysis process was carried out using a catalyst. The type of catalyst used is an acid catalyst (H_2SO_4).

Hydrolysis is a chemical reaction between water and another substance that produces new substances and causes a solution to decompose using water. Hydrolysis reactions are generally endothermic reactions (requires heat). The process of hydrolysis of cellulose follows the following equation: $(C_6H_{10}O_5)n + n\ H_2O \rightarrow n\ C_6H_{12}O_6$.

Hydrolysis using an acid catalyst is influenced by temperature, acid concentration, and the material to be hydrolyzed. The effect of temperature on the rate of reaction follows the Arrhenius equation: the higher the temperature, the faster the reaction [3]. The effect of acid concentration in the hydrolysis process, the higher the acid concentration, the higher the sugar content after going through the hydrolysis stage [4]. The material to be hydrolyzed depends on the amount of cellulose contained in the material, the more cellulose contained in the material, the greater the conversion of cellulose to glucose.

This research is intended to produce glucose through acid hydrolysis of water hyacinth plants with variations in temperature, acid concentration, and ingredients (parts of water hyacinth: stems, leaves, and mixture) to obtain the best acid hydrolysis conditions. Determination of the best acid hydrolysis conditions is reviewed. from the value of the highest glucose concentration which was analyzed by the refractometer method.

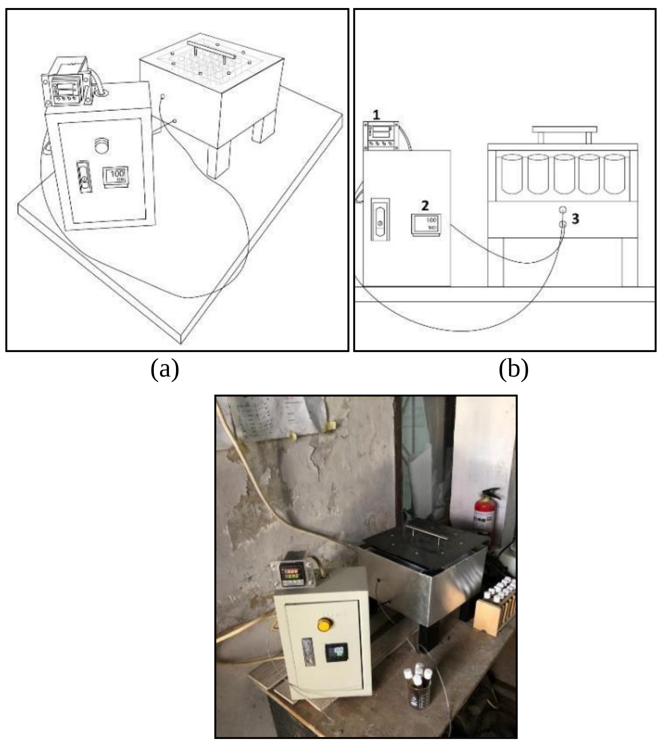
METHODOLOGY

Approach

Glucose production from water hyacinth is taken from Cirata Reservoir located in West Bandung Regency, West Java Province. The conversion of cellulose to glucose is carried out through an acid hydrolysis process. Acid hydrolysis was carried out at temperatures of 90°C, 100°C, and 110°C. The required hydrolysis time is 90 minutes. The type of acid used is H_2SO_4 with concentrations of 2%, 4%, 6%, 8%, 10%, and 12%. The materials used are water hyacinth stems, leaves, and a mixture. The result of the hydrolysis is determined by the concentration of glucose through refractometer analysis.

Tools and Materials

The main equipment used is an acid hydrolysis device. The materials used were water hyacinth, 0.5 M NaOH, aqua dest, and H_2SO_4 as shown in Figure 1.



(a) (b)

(c)

FIGURE 1. Acid Hydrolysis Equipment (a) Equipment Schematic Top View (b) Equipment Schematic Side View (c) Tool Photo

Research Procedure

Raw Material Preparation Stage

The raw material preparation stage in this study aims to prepare raw materials that will be used during the research process, including water hyacinth which is used as a substrate, and water as a supporting component in the study. The initial stage is the process of cutting the water hyacinth substrate into smaller sizes. After the size becomes smaller, the next process is delignification by adding 0.5 M NaOH into the water hyacinth then let stand for 90 minutes. The solution was filtered, then the residue of the solution which was lignin-free water hyacinth was washed with distilled water until the pH of the residue was neutral. Water hyacinth is dried which aims to reduce the water content in water hyacinth to obtain the same ratio. After drying, the dried water hyacinth is blended until it becomes a powder. Then the cellulose content was analyzed at the Center for Pulp and Paper.

Acid Hydrolysis Stage

First, make a solution of H2SO4 with concentrations of 2%, 4%, 6%, 8%, 10%, and 12%. 4 grams of dry water hyacinth was put into a test tube with a screw cap and then 9 mL of H2SO4 solution was added. The solution was hydrolyzed at various temperatures of 90ºC, 100ºC, and 110ºC for 90 minutes. The hydrolysis process results were analyzed for glucose content by the refractometer method.

RESULTS AND DISCUSSION

Raw Material Preparation

The delignification process will remove the lignin content in water hyacinth so that the conversion of cellulose to glucose during the hydrolysis process is getting better. Lignin dissolved in NaOH solution is marked with black color in the solution which is called black liquor. The results of the remaining delignification process can be seen in Figure 2.

FIGURE 2. Results of the Delignification Process Immersion Remaining

Water hyacinth was also obtained from each part of the water hyacinth. The water content contained in the water hyacinth stem was 88%, the leaf was 69%, and the mixture (stem and leaf) was 78.5%. The dried stems and leaves were analyzed for their cellulose content at the Center for Pulp and Paper located in Dayeuhkolot, Bandung, West Java. The material used for cellulose analysis requires as much as 1 kg of dry matter, where the composition is 650 g of dry stems and 350 g of dry leaves. The method for analyzing the cellulose content used the ASTM D 1103-60 method. The cellulose content of dried water hyacinth stems and leaves according to the test results report was 50.15% (w/w).

Acid Hydrolysis

Acid hydrolysis was carried out by adding sulfuric acid (H_2SO_4) with concentrations of 2%, 4%, 6%, 8%, 10%, and 12%. Acid hydrolysis was carried out on a variety of stem, leaf, and mixed materials. Each material with a mesh size of 40/60 was put into a 4 gram test tube and 9 mL of sulfuric acid was added. Acid hydrolysis is carried out in a closed heater using a halogen lamp as a heat source. Acid hydrolysis was carried out for 90 minutes at 90˚C, 100˚C, and 110˚C. The use of a small acid concentration aims to avoid corrosive properties and save costs because there is no need to use expensive metal equipment.

Effect of Temperature on Glucose Concentration Result of Acid Hydrolysis

In this study, an analysis of the glucose content of the suspension resulting from the acid hydrolysis process was carried out using a refractometer. The results of the analysis of the effect of temperature on water hyacinth glucose levels are shown in the following Figure 3 - 5.

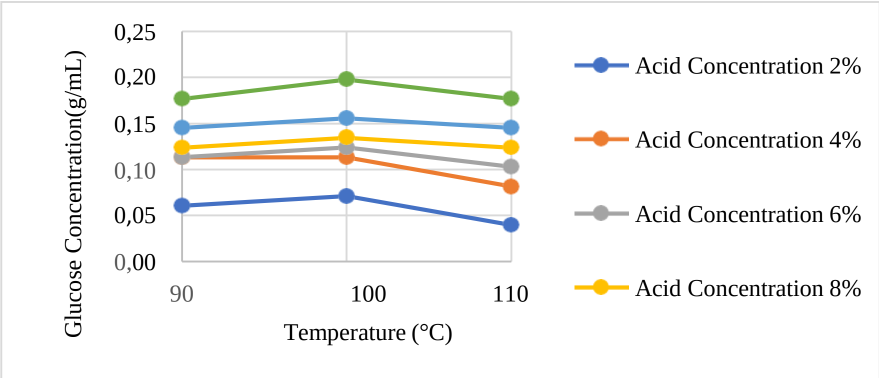


FIGURE 3. The Curve of the Effect of Temperature on the Concentration of Glucose from Acid Hydrolysis in Stem Material

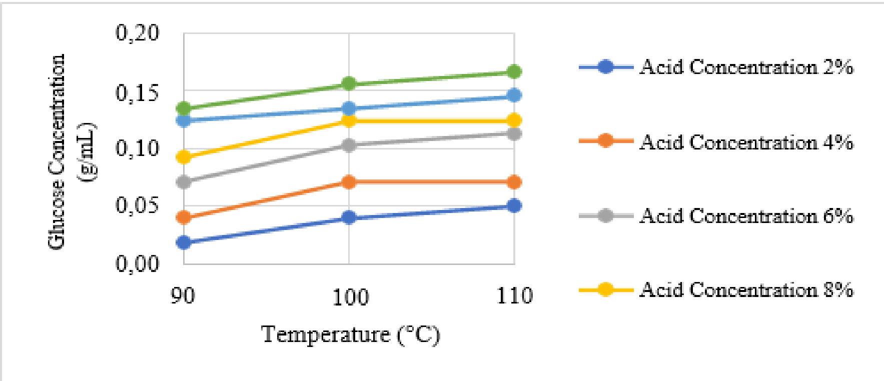


FIGURE 4. The Curve of the Effect of Temperature on the Concentration of Glucose from Acid Hydrolysis in Leaf Material

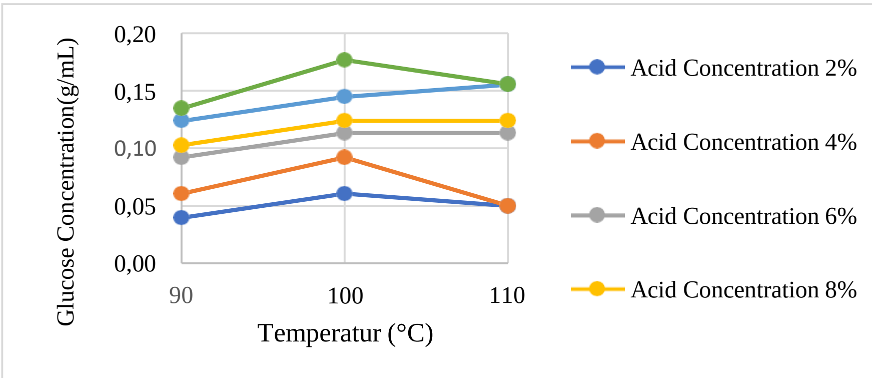


FIGURE 5. The Curve of the Effect of Temperature on the Concentration of Glucose from Acid Hydrolysis in Stem-Leaf Material

Water hyacinth glucose levels were analyzed after going through the acid hydrolysis process using H2SO4. The effect of temperature on the rate of a reaction follows the Arrhenius equation: the higher the temperature, the faster the reaction proceeds. It can be seen in the curves of Figure 3 and Figure 4 that there is a difference between stem and leaf material. The stem material has a higher cellulose content than the leaf material. Therefore, the cellulose converted to glucose will be greater than the leaf material, the maximum limit for acid hydrolysis with stem material at a temperature of 100ºC, because if the temperature is more than 100ºC, the glucose formed will undergo a caramelization reaction. The caramelization reaction occurs in glucose generally at a temperature of 160ºC, but the caramelization reaction can be accelerated if the glucose is in an acidic state. By controlling the level of acidity (pH), the rate of the caramelization reaction can be changed. The rate of caramelization can be accelerated under acidic conditions (especially pH below 3) [5]. In contrast to leaves, leaves have less cellulose content than stems, so the cellulose converted to glucose is also small, which in leaf material the glucose concentration produced is constant and some increases with increasing hydrolysis temperature. The leaf material tends not to experience a caramelization reaction. Likewise, the mixed stem and leaf material obtained various results as shown in Figure 5.

Effect of Acid Concentration on Glucose Concentration Result of Acid Hydrolysis

The results of the analysis of the effect of acid concentration on water hyacinth glucose levels are shown in the following Figure 6 – 8.

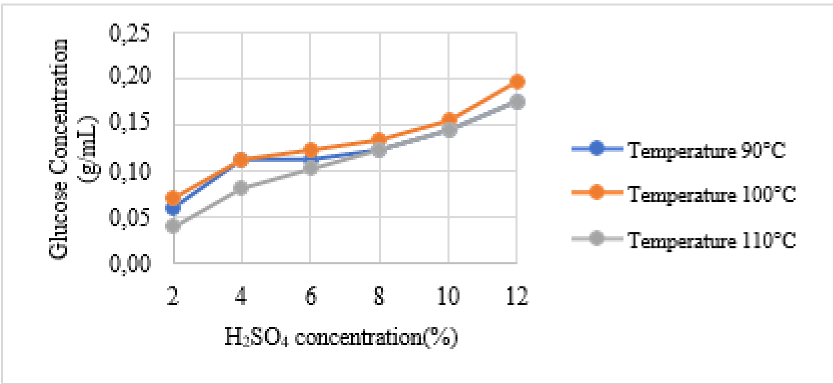


FIGURE 6. The Curve of the Effect of Acid Concentration on Glucose Concentration of Acid Hydrolysis Results in Stem Material

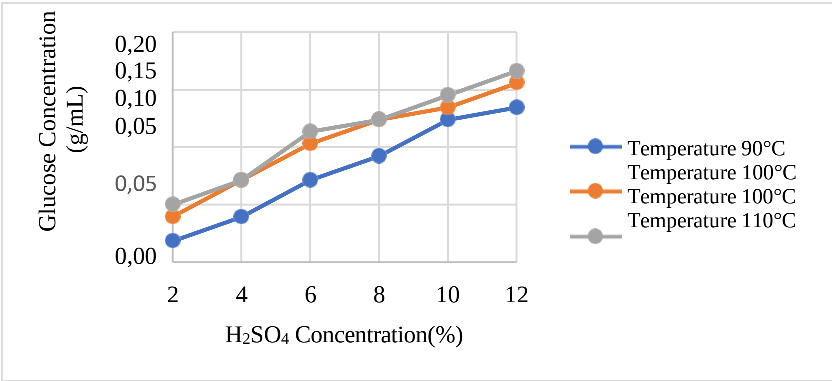


FIGURE 7. The Curve of Effect of Acid Concentration on Glucose Concentration of Acid Hydrolysis Results on Leaf Material

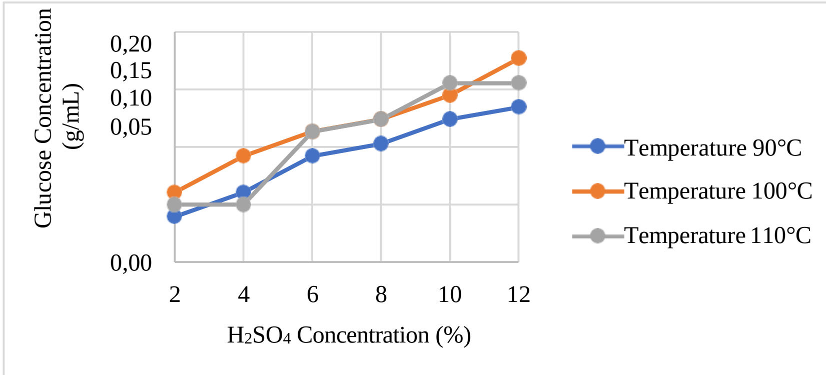


FIGURE 8. The Curve of Effect of Acid Concentration on Glucose Concentration of Acid Hydrolysis Results on Stem-Leaf Material

Figure 6, Figure 7, and Figure 8 show the effect of acid concentration on the glucose concentration resulting from hydrolysis. According to the theory, the use of high concentrations of acid will provide high sugar content after going through the hydrolysis stage [4]. From Figure 6, Figure 7, and Figure 8 it is known that the hydrolysis process of water hyacinth using H_2SO_4 solution accompanied by closed heating at temperatures of 90˚C, 100˚C and 110˚C produces different glucose levels for the six concentrations used. In the hydrolysis stage, the sample is heated by heating using hot steam. The resulting hot steam will increase the ability of H_2SO_4 to break down cellulose into glucose. In addition, the water content contained in the raw materials also affects the glucose levels produced, the less water content contained in water hyacinth, the higher the glucose levels obtained. It can be seen that the higher the concentration of acid used in the hydrolysis process, the higher the concentration of glucose produced. This is by the existing theory. Judging from the curves in Figure 6, Figure 7, and Figure 8, the concentration of glucose resulting from hydrolysis increases with the amount of acid concentration. This is because, at higher acid concentrations, more cellulose breaks down into glucose. In this study, the sulfuric acid concentration of 12% is the maximum concentration that can be used for the acid hydrolysis process. The lower the pH in the acid hydrolysis process, the faster the caramelization reaction.

The caramelization reaction will inhibit the conversion of cellulose to glucose. If caramelization occurs, the concentration of glucose formed will decrease. Figure 6, Figure 7, and Figure 8 show the effect of acid concentration on the concentration of glucose resulting from hydrolysis. According to the theory, the use of high concentrations of acid will provide high sugar content after going through the hydrolysis stage [4]. From Figure 6, Figure 7, and Figure 8 it is known that the hydrolysis process of water hyacinth using H2SO4 solution accompanied by closed heating at temperatures of 90˚C, 100˚C and 110˚C produces different glucose levels for the six concentrations used. In the hydrolysis stage, the sample is heated by heating using hot steam. The resulting hot steam will increase the ability of H_2SO_4 to break down cellulose into glucose. In addition, the water content contained in the raw materials also affects the glucose levels produced, the less water content contained in water hyacinth, the higher the glucose levels obtained. It can be seen that the higher the concentration of acid used in the hydrolysis process, the higher the concentration of glucose produced. This is by the existing theory. Judging from the curves in Figure 6, Figure 7, and Figure 8, the concentration of glucose resulting from hydrolysis increases with the amount of acid concentration. This is because, at higher acid concentrations, more cellulose breaks down into glucose. In this study, the sulfuric acid concentration of 12% is the maximum concentration that can be used for the acid hydrolysis process. The lower the pH in the acid hydrolysis process, the faster the caramelization reaction. The caramelization reaction will inhibit the conversion of cellulose to glucose. When caramelization occurs, the concentration of glucose formed will decrease.

Effect of Material on Glucose Concentration Result of Acid Hydrolysis

The results of the analysis of the effect of acid concentration on water hyacinth glucose levels are shown in the following Figure 9 – 11.

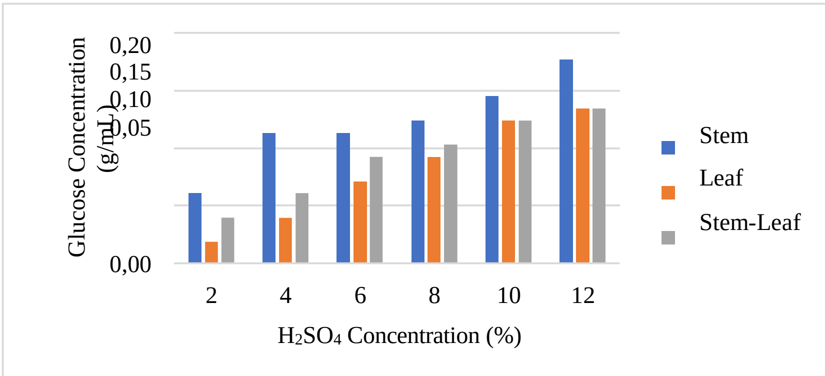


Figure 9. Diagram of the Effect of Materials on Glucose Concentration of Acid Hydrolysis at 90°C Temperature

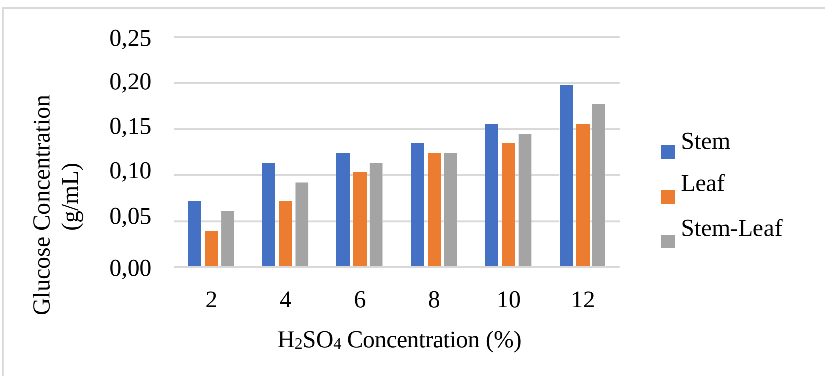


Figure 10. Diagram of the Effect of Materials on Glucose Concentration of Acid Hydrolysis at 100°C Temperature

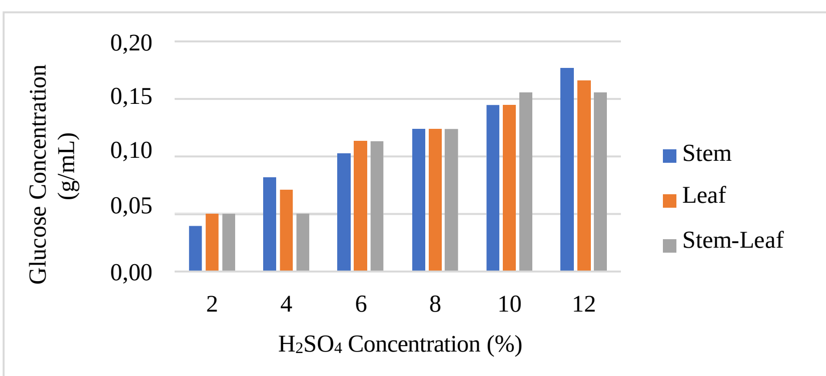


Figure 11. Diagram of the Effect of Materials on Glucose Concentration of Acid Hydrolysis at 110°C Temperature

In Figures 9 and 10, it can be seen that the stem material at the same temperature and acid concentration of glucose produced from acid hydrolysis has a higher glucose concentration than the leaf and mixed material (stem and leaf). This can happen because the stems contain more cellulose than the leaves. The cavity of the water hyacinth stem is the same as that of a banana midrib, so it can be assumed that

The cellulose content in water hyacinth stems is greater than in the leaves. The cellulose content in the banana midrib is 83.3% while the banana leaf has a cellulose content of 10.85% [6]. However, there are exceptions or differences at a temperature of 110ºC where the curve in Figure 11 can be seen that the results of the acid hydrolysis glucose concentration from the stem material are not always higher, this is due to the caramelization process. The caramelization reaction will inhibit the conversion of cellulose to glucose. When caramelization occurs, the concentration of glucose formed will decrease.

The cellulose contained in dry water hyacinth which will be used as raw material for the acid hydrolysis process has a composition of 50.15% according to the analysis using the ASTM D 1103-60 method. From the experiments that have been carried out, it was obtained that the glucose with the highest concentration of 0.178 g/mL was 9 mL from acid hydrolysis with an acid concentration of 12% at 100°C and the material used was the stem. The results of the mass conversion of water hyacinth, dry water hyacinth mass, and cellulose mass to the results of the best acid hydrolysis process of each material can be seen in Table 1, Table 2, and Table 3.

TABLE 1. Results of Mass Conversion of Wet Water Hyacinth, Dry Water Hyacinth Mass and Cellulose Mass to the Result of Acid Hydrolysis Process on Stem Material (Hydrolysis Temperature 100°C and Acid Concentration of 12%)

Stem Moisture Content	Wet Stem Mass (g)	Dry Stem Mass (g)	Glucose Concentration (g/mL)	Glucose Mass (g)	% Glucose Yield /Ingredient Wet Stem	% Glucose Yield /Ingredient Dried Stem
88%	33,33	4	0,1978	1,78	5,34%	44,51%

TABLE 2. Mass Conversion Result of Wet Water Hyacinth, Dry Water Hyacinth Mass and Cellulose Mass to Result of Acid Hydrolysis Process on Leaf Material (Hydrolysis Temperature 110°C and Acid Concentration of 12%)

Stem Moisture Content	Wet Stem Mass (g)	Dry Stem Mass (g)	Glucose Concentration (g/mL)	Glucose Mass (g)	% Glucose Yield /Ingredient Wet Stem	% Glucose Yield /Ingredient Dried Stem
69%	12,90	4	0,1662	1,50	11,59%	37,40%

TABLE 3. Mass Conversion Results of Wet Water Hyacinth, Dry Water Hyacinth Mass and Cellulose Mass to the Result of Acid Hydrolysis Process on Stem-Leaf Material (Hydrolysis Temperature 100°C and Acid Concentration of 12%)

Stem-Leaf Water Content	Wet Stem-Leaf Mass (g)	Dry Stem-Leaf Mass (g)	Cellulose Mass (g)	Glucose Concentration (g/mL)	Glucose Mass (g)	% Glucose Yield /Ingredient Dried Stem	% Glucose Yield /Ingredient Dried Stem	% Yield Glucose/ Cellulose
78,50%	18,60	4	2,01	0,1767	1,59	8,55%	39,77%	79,30%

CONCLUSION

From the research that has been done, the best acid concentration value in the acid hydrolysis process is 12% acid concentration at a temperature of 100°C and the material used is stem glucose concentration is 0.1788 g/mL. The %yield value of glucose weight/weight of water hyacinth was 5.34% and the %yield by weight of glucose/weight of dry water hyacinth was 44.51%.

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REFERENCES

1. Nuryana, R., *Eceng Gondok (Eichornia crassipes),* eprints.polsri.ac.id., (2016). (Accessed on December 8, 2020)
2. Permatasari, H. R., et.al., "Effect of Concentration of H_2SO_4 and NaOH on Delignification of Bamboo Powder (Gigantochloa Apus)", *Jurnal Penelitian Pendidikan Kimia – Kajian Hasil Penelitian Pendidikan Kimia,* Vol. 1 (2): 131 – 140, (2014).
3. Groggins, P. H., *Unit Processes in Organic Synthesis*, 5^{th} ed., p. 775-777. New York: McGraw-Hill Book Company, (1958).
4. Hamelinck, C.N., van Hooijdonk, G., & Faaij, A.P.C., "Ethanol from Lignocellulosic Biomass: Techno-Economic Performance in Short-, Middle- And Long-Term", *Biomass Bioenergy*, p. 384–410, (2005).
5. Dennis D., *Food Chemistry*, (1993).
6. Bahri, S., "Making Pulp from Banana Stems", *Journal of Chemical Technology Unimal*, Vol. 4 (2): 36-50, (2015).