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Glucose Syrup from Purple Sweet Potatoes (*Ipomoea batatas L. Poir*) using Acid Hydrolysis Method

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1. INTRODUCTION

Food and drink are basic needs for humans to survive. In the beginning, humans could only process food very simply from food ingredients that could be consumed. Humans have a desire to find new flavors, new processing methods, and even materials that can be processed and consumed. This is a good thing because then humans innovate in the food sector. Innovation in the food sector also needs to pay attention in terms of the benefits of food and beverages made. The expected benefits are not only in terms of flavors that people like but without them knowing the content of the food. Therefore, it is necessary to innovate processed foods and beverages that are superior in content and benefits [1].

Indonesia is an agricultural country that has a tropical climate, so the majority of its people make their living as farmers. This is because Indonesia is rich in natural resources that can be utilized by people from the lowlands to the highlands in remote areas of the country [2]. One thing that is very easy to find in this tropical climate is tuber plants such as sweet potatoes. Among the various kinds of sweet potatoes that can be processed into various types of food, one of them is purple sweet potato (*Ipomoea batatas L. Poir*). Processed purple sweet potatoes are often found, especially in Tawangmangu, Karanganyar, Central Java [3].

The purple sweet potato plant is one type of root tuber plant from the total tuber genus that grows in Indonesia. This plant grows in tropical areas that have humid and hot conditions. Ideally for sweet potato plants grow in an environment with a temperature of 21-27°C with rainfall of 750-1500 mm per year and almost suitable in all regions across the country [4]. Purple sweet potato type *Ipomoea batatas L. Poir* has a fairly intense purple color in the flesh of the sweet potato so that it has its own charm [5]. This purple sweet potato also has abundant nutritional content, namely as much as 150.7 mg/100 g anthocyanins, 1.1% fiber, 18.2% starch, 0.4% reducing sugar, 0.6% protein, 0.70 mg/100 g iron, and 20.1 mg/100 g vitamin C [6].

Indonesian people have long used purple sweet potatoes but still processed traditionally. Purple sweet potato is a source of starch which has the potential to be developed into a functional food [7]. Due to its various benefits and contents, purple sweet potato was chosen as processed sugar because it has a lower calorie content and glycemic index than sugarcane [8]. However, there are still many people who do not know that purple sweet potatoes can also be used as a sweet taste enhancer by processing them into glucose syrup.

Glucose syrup is different from liquid sugar. Liquid sugar can come from various sources such as sugar cane, sugar beet, coconut, palm, and others which contain sucrose or a mixture of simple sugars. Meanwhile, glucose syrup is generally produced from starch derived from corn, cassava, wheat, or sorghum through a hydrolysis process [9]. Glucose syrup contains D-glucose, maltose, and D-glucose polymers made through starch hydrolysis [10]. In general, hydrolysis is a process of separating chemical bonds using water. The process of starch hydrolysis is the breakdown of amylum molecules into simpler constituent components such as dextrin, maltotriose, maltose, and glucose [11]. The more perfect hydrolysis process can be indicated by the higher dextrin equivalent (DE) value, which means that if the DE value reaches 100, the hydrolysis process produces 100% reducing sugar [12].

Glucose syrup began to be widely used in the food and beverage industry because it has several advantages. Liquid glucose syrup is widely used in the food, beverage, and fermented product industries because of its stable properties, inhibited crystallization, increased shelf life, and natural taste [13, 14].

Research on making glucose syrup from purple sweet potatoes has been carried out through hydrolysis using hydrochloric acid [15], sulfuric acid [16], and citric acid [17]. Glucose syrup synthesis from Cilembu Sweet Potato has been carried out through acid hydrolysis using hydrochloric acid with various concentrations and different hydrolysis times. The best results were obtained from acid hydrolysis for 60 minutes with a hydrochloric acid concentration of 0.4 N [18]. Similar research has also been conducted using sweet potato (Ipomoea Batatas L.) as raw material [19]. [19]. In this study, glucose syrup was made from purple sweet potatoes (*Ipomoea batatas L. Poir*) through the acid hydrolysis method. Hydrolysis was carried out using food-grade citric acid with various concentrations. The citric acid used is a technical acid that can be easily obtained in the market. The purpose of this study was to determine the ability of food-grade citric acid to hydrolyze and to determine the characteristics of the resulting glucose syrup compared to SNI-01-2978-1992. Standard parameters from SNI 01-2978-1992 include organoleptic aspects (odorless, sweet taste, colorless), water content (maximum 20%), ash content (maximum 1%), reducing sugar content (minimum 30%), and starch content (negative). The toxicity of glucose syrup was also checked using the Brine Shrimp Lethality Test (BSLT) [20].

2. MATERIALS AND METHODS

The tools used in this experiment are knife, grater, mortar and pestle, beaker glass, measuring flask, volume pipette, Erlenmeyer, digester, test tube, aerator, porcelain cup, porcelain crucible, furnace, and oven.

The main materials used in this experiment are purple sweet potatoes (*Ipomoea batatas L. Poir*) obtained from the plantations in Tawangmangu, Karanganyar and surrounding areas. The supporting materials used are technical citric acid ($C_6H_8O_7$), baking soda (NaHCO₃), sodium chloride (NaCl), aquadest, and lugol which can be purchased commercially on the market. For analysis, sulfuric acid (H_2SO_4) was purchased from EMSURE at a concentration of 98%. The flow chart of the method used in this study can be seen in Figure 1 and Figure 2.

2.1 Preparation of Purple Sweet Potato Flour

The purple sweet potatoes were peeled and washed clean then grated. The starch was taken by adding a little water and then squeezing it until the sweet potato no longer releases starch juice. Starch was obtained from the precipitate formed after leaving it for 6 hours. Then, the precipitate was separated and dried in an oven at 60°C for 24 hours. The dry flour is ground using a mortar and then sifted.

2.2 Preparation of Glucose Syrup from Purple Sweet Potato

Glucose syrup is prepared by mixing 25 grams of dry flour into 75 ml of boiling water. Then 15 ml of citric acid (0.2 N, 0.4 N, 0.6 N, 0.8 N, 1 N) was added to each sample. The mixture was heated to 121°C for 60 minutes. The pH was checked using pH indicator stick and neutralized by adding 0.05 N NaHCo₃ until the pH approached 7. The glucose syrup formed was then centrifuged for 60 minutes at 8 rpm to separate the existing sediment. The supernatant was separated and concentrated by heating the glucose syrup on a hot plate at 100°C for 20 minutes.

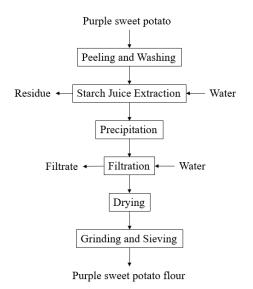


Figure 1. Flow chart of purple sweet potato flour preparation

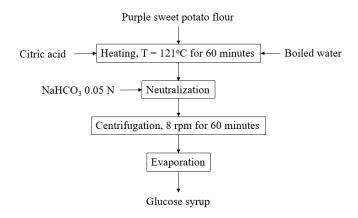


Figure 2. Flow chart of glucose syrup preparation from purple sweet potato

2.3 Toxicity Testing

The toxicity testing was done using the Brine Shrimp Lethality Test (BSLT) [21]. The toxicity test begins by preparing Artemia Salina Leach larvae by incubating the eggs in 100 mL of NaCl solution (0.4 grams/ 100 mL distilled water) and aerating them for 24 – 48 hours. Next, a standard sample solution with a concentration of 2.000 ppm was made by dissolving 40 mg of glucose syrup in 20 ml of NaCl solution.

The test solution was prepared by diluting the standard solution to 100, 50, 25, and 12.5 ppm. NaCl solution (0 ppm) was used as a control. Then 10 shrimp larvae that had hatched were put into each 6 ml of test solution and observed for 24 hours. LC_{50} is obtained by counting dead shrimp larvae at intervals of 1, 2, 3, 4, 5, 6, 12, 18, and 24 hours using Equation (1).

$$\text{\%Death} = \frac{\text{number of dead larvae}}{\text{number of test larvae}} \times 100\%$$
(1)

Then the results of %Death are plotted based on concentration and the lethal concentration is looked for when %death = 50% as the LC₅₀ value. Sangi [20] reported that a product is said to be toxic if it can cause the death of 50% of test animals at a concentration of <1,000 ppm.

2.4 Water Content Analysis

Water content was measured by placing the sample in an oven for 3 hours at 105°C and placing it in a desiccator for 15 minutes [22]. Repeat until the sample weight is constant and the water content can be measured using Equation (2).

Water content = $\frac{\text{Initial sample mass} - \text{final sample mass}}{\text{initial sample mass}} \times 100\%$ (2)

2.5 Ash Content Analysis

Ash content was measured by placing the sample in a furnace for 3 hours at 500°C. The ash content can be measured using Equation (3) [23].

Ash content =
$$\frac{\text{Initial sample mass} - \text{final sample mass}}{\text{initial sample mass}} \times 100\%$$
 (3)

2.6 Reducing Sugar Content Analysis

Reducing sugar content was calculated using the du Bois method [24, 25]. Sample preparation was carried out by taking 1 ml of glucose syrup and diluting it up to $2000\times$. Then 0.1 ml of phenol was added to 2 ml of the diluted sample and 5 ml of sulfuric acid (H₂SO₄) was also added. Then, the solution was placed in a shaker incubator for 20 minutes at a speed of 125 rpm. The sample was tested using a UV-visible spectrophotometer (Thermo Scientific Genesys 20) at a wavelength of 490 nm. The reading results were then plotted on a standard curve to determine the reducing sugar content.

2.7 Strach Content Analysis

The glucose syrup sample that has been in the oven is placed in a test tube. Then distilled water is added to the test tube and shaken until homogeneous. The starch content was tested qualitatively by adding 1-3 drops of Lugol's solution and observing the color change. If the color changes to dark blue or purple, the sample is positive for starch [26, 27].

2.8 Organoleptic Testing (Odor, Taste, and Color)

Organoleptic tests can be carried out on a small scale in a laboratory setting with at least 1 to 3 panelists [28]. Panelists give an assessment based on predetermined criteria. Organoleptic value assessment is carried out for odor, taste, and color. The organoleptic test scale can be seen in Table 1.

Table 1. Organoleptic Test Scale						
Scale	Odor	Taste	Color			
1	Very smelly	Not very sweet	Brown			
2	Smell	Not sweet	Light brown			
3	Sight odor	Slightly sweet	Brownish-yellow			
4	No odor	Sweet	Light yellow			
5	Very odorless	Very sweet	Colorless			

3. RESULTS AND DISCUSSION

This research began with preparing the main ingredient, namely purple sweet potato, until glucose syrup was formed which is divided into 2 stages: preparing flour and preparing glucose syrup by acid hydrolysis using citric acid of various concentrations. The resulting glucose syrup was then characterized and compared with the Indonesian national standard (SNI-01-2978-1992). The tests carried out were toxicity tests, water content tests, ash content tests, sugar content tests, and qualitative starch content tests. Apart from that, each sample is subjected to organoleptic tests in the form of smell, color, and taste tests. Following are the results of each test that has been carried out.

3.1 Toxicity Testing Result

The toxicity test conducted in this study was intended to determine the toxic potential of the purple sweet potato glucose syrup produced. The toxicity test in this study used the Brine Shrimp Lethality (BSLT) method by determining the LC_{50} value to determine the toxicity level of glucose syrup from purple sweet potato. The results of the toxicity test using the Brine Shrimp Lethality Test (BSLT) method with 3 repetitions of testing on glucose

syrup from purple sweet potato obtained average results as in Table 2.

Citric Acid	Concentration	Shrimp	LC ₅₀ (ppm)	Description
Concentrat	(ppm)	Death (%)		
ion (N)				
0,2	0	30	1,391.553	Non-toxic
	12.5	33		
	25	33		
	50	37		
	100	43		
0.4	0	13	14,138.391	Non-toxic
	12.5	17		
	25	20		
	50	27		
	100	30		
0.6	0	33	1,112.449	Non-toxic
	12.5	37		
	25	40		
	50	43		
	100	43		
0.8	0	37	4,857.356	Non-toxic
	12.5	37		
	25	40		
	50	40		
	100	43		
1	0	23	9,997.697	Non-toxic
	12.5	27		
	25	30		
	50	33		
	100	37		

Table 2. Larva Mortality on Purple Sweet Potato Glucose Syrup with Variations of Citric Acid Concentration

According to Sangi, reported that a material or product shows toxic activity in toxicity testing if it can cause the death of 50% of test animals at a concentration of < 1.000 ppm [20]. In the table presented above, it is found that in the observation for 24 hours in each variation of critic acid concentration, the LC_{50} value is more than 1.000 ppm. This proves that glucose syrup from purple sweet potato does not have toxic potential so it is safe for consumption.

3.2 Water Content Analysis Result

In testing the water content of glucose syrup from purple sweet potato, data is obtained as in Figure 3. Water content will increase as the concentration of citric acid increases. This is because the higher the concentration of citric acid used, the more NaHCO₃ solution is added in the neutralization process so that the water contained in the sample increases and causes the glucose syrup to dilute. The water content will affect the viscosity or viscosity of the glucose syrup. The lower the water content obtained, the higher the viscosity of the glucose syrup so that the resulting glucose syrup will be thicker [18]. The highest water content was in the variation of 1 N citric acid concentration of 19,92% (%mass) and the lowest water content was in the variation of 0,2 N concentration of 17.34%.

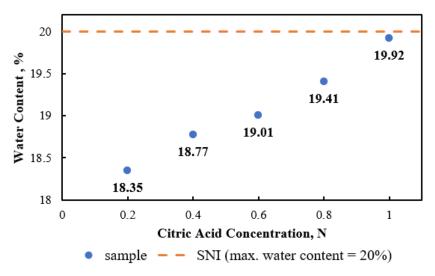


Figure 3. The result of water content analysis compared to SNI

According to SNI 01-2978-1992, the maximum water content in glucose syrup is 20%. In this study, the water content contained in glucose syrup from various variations of citric acid concentration did not exceed the limit in SNI so it can be concluded that the glucose syrup product in this study has met SNI.

3.3 Ash Content Analysis Result

In testing the ash content of glucose syrup from purple sweet potatoes, data was obtained whose calculation results were presented in Figure 2. The results of this analysis did not show a trend as in the water content analysis. It can be seen that the ash content increases from a citric acid concentration of 0.2 N to 0.4 N. But it drops drastically at a citric acid concentration of 0.6 N and the content shows an increase up to a citric acid concentration of 1 N. 0.4 N. The highest ash content at the variation in 0.2 N citric acid concentration was 14.63% (%mass) and the lowest ash content in the 0.6 N citric acid concentration was 7.95%.

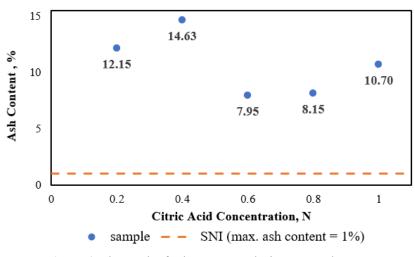


Figure 4. The result of ash content analysis compared to SNI

According to SNI 01-2978-1992, the maximum ash content in glucose syrup is 1%. From Figure 4 it can be seen that the ash content at all citric acid concentrations exceeds the standard limit. The ash content that did not meet the requirements could be due to the addition of a greater concentration of citric acid so that the glucose syrup produced will be more acidic which results in the addition of NaHCO₃ solution in the neutralization process. High ash content can also be caused by insufficient physical treatment when making glucose syrup so it needs to be filtered again so that the ash content meets standards.

3.4 Reducing Sugar Content Analysis Result

The reducing sugar content test was carried out using a UV-Vis spectrophotometer with a wavelength of 490 nm. Before testing, the sample was diluted $2000 \times$ and the test was done $3 \times$ repetitions. Based on the test results, it can be seen that the concentration of citric acid affects the concentration of reducing sugar. As seen in Figure 5, it was found that the sugar content increased along with the concentration of citric acid up to a concentration of 0.4 N and decreased at concentrations greater than 0.4 N. This decrease is possible because the NaHCO₃ solution needed for neutralization is more, so the sugar content is lower. The test results show the best sugar content in samples with citric acid concentration of 0.4 N.

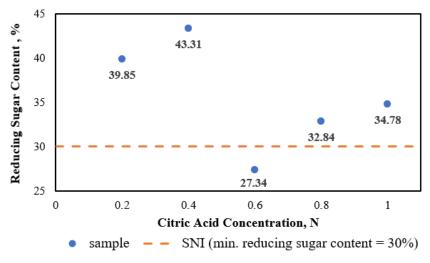


Figure 5. The result of reducing sugar content analysis compared to SNI

Based on the results of the percentage of reducing sugar from purple sweet potato glucose syrup with the highest sugar content with 0.4 N citric acid concentration of 43.31%. This shows that the highest percentage of reducing sugar from purple sweet potato glucose syrup with 0,4 N citric acid concentration meets the standard standards that have been determined by referring to SNI 01-2978- 1992 reducing sugar standards of at least 30%.

3.4 Starch Content Analysis Result

Starch is a polysaccharide carbohydrate that is widely found in flour derived from tubers. From the qualitative starch analysis that has been carried out on glucose syrup samples with varying concentrations of citric acid, positive results were obtained containing starch and negative did not contain starch. Samples that positively contain starch are characterized by a change in color to purple when dripped with Lugol's solution. Samples with 0.2 N citric acid concentration positively contain starch while samples with 0.4 N, 0.8 N, and 1 N concentrations negatively do not contain starch as seen in Table 3.

Table 3. Starch Content Analysis Data					
Citric Acid	Color Changes	Starch Content			
Concentration (N)					
0.2	Yes	Positive			
0.4	No	Negative			
0.6	No	Negative			
0.8	No	Negative			
1	No	Negative			

Glucose syrup samples that do not change color when tested with Lugol's solution, it can be stated that the raw material of purple sweet potato starch flour has been completely hydrolyzed [25]. While samples that show positive results indicate that there is still starch that has not been hydrolyzed. According to SNI 01-2978-1992, the standard of glucose syrup does not contain starch so that glucose syrup with the addition of 0,2 N citric acid concentration does not meet the requirements.

3.5 Organoleptic Testing Result

This organoleptic test was conducted as an initial survey of the products produced so that the number of respondents used was small and was not processed statistically. The results of the organoleptic tests that have been carried out can be seen in Table 4.

Table 4. Organoleptic Testing Data

Citric Acid	Panelist —	Parameters Tested		
Concentration (N)		Odor	Taste	Color
0,2	1	No odor	Not sweet	Brownish-yellow
	2	No odor	Not sweet	Brownish-yellow
	3	No odor	Not sweet	Brownish-yellow
0,4	1	No odor	Not sweet	Brown
	2	No odor	Not sweet	Brown
	3	No odor	Not sweet	Brown
0,6	1	No odor	Not sweet	Brown
	2	No odor	Not sweet	Brown
	3	No odor	Not sweet	Brown
0,8	1	No odor	Not sweet	Brown
	2	No odor	Not sweet	Brown
	3	No odor	Not sweet	Brown
1	1	No odor	Not sweet	Brown
	2	No odor	Not sweet	Brown
	3	No odor	Not sweet	Brown

Based on the organoleptic test results of purple sweet potato glucose syrup with varying concentrations of citric acid, it show that increasing the concentration of citric acid added has no significant effect on the odor, color, and taste of the glucose syrup. However, these results do not meet the Indonesian National Standards for glucose syrup because the results of making glucose syrup from purple sweet potato have the color as presented in Table 4. The resulting taste contains a slightly sour taste due to the influence of the use of citric acid so it can make the taste of glucose syrup from purple sweet potato not sweet [29], but the odor produced by glucose syrup from purple sweet potato is odorless and still meets the quality standards of glucose syrup (SNI 01-2978-1992).

4. CONCLUSION

Based on the data obtained from the study, it can be concluded that glucose syrup from purple sweet potatoes made by acid hydrolysis method using citric acid has not met the quality standards in its entirety and still requires much improvement. The best results were obtained in glucose syrup made by hydrolysis with citric acid concentration of 0.4 N. The water, reducing sugar, and starch content in that sample were following SNI 01-2978-1992, while the ash content was still far above the standard. In the organoleptic test, only the odor met with standard, while the taste was not sweet even though the reducing sugar content was above the standard. However, the results of the toxicity test on all the samples showed that the lethal concentration or LC_{50} value exceeded 1,000 ppm, which means that purple sweet potato glucose syrup has no toxic potential so it is safe for consumption. However, the making of glucose syrup through acid hydrolysis with food-grade citric acid was almost successfully done. Improvements for further research can be made by increasing the sweetness and reducing the ash content in the resulting glucose syrup so that it can meet quality standards.

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