Characterization of Sago Starch and Study of Liquefication Process on High Fructose Syrup Production

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Abstract :

Sago starch contains high carbohydrate and found in large quantities in Indonesia. However, the utilization of sago starch have not been up to. In this study, hopefully sago starch can be used as one of the alternative sweetener called high fructose syrup. The study was divided into two, named characterization study and liquefication stage study. Characterization of the starch was carried out by the determination of carbohydrate content, water content, amylose content of starch, starch gelatination temperature, swelling and amylose leaching, and carbohydrat leaching. The liquefication stage study was conducted by varying enzyme and sago starch suspense concentration in producing dextrin to obtain the best value of concentration. The result of the characterization studies shows the value of carbohydrate contained in starch is 84,78% with the value of amylose concentration is 25,65%, water content of 14,1-17,2%, gelatinization temperature of 76-86^{o}C. Liquefication stage study represents that the highest yield is given by sago starch concentration of 25% w/v with α-amylase enzyme concentration of 0,1% v/w and liquefication time length process of two hours.

Keywords: dextrin; liquefication process; sago starch

1. Introduction

Today, the need of sugar in Indonesia is estimated to reach 4,1 million tons per year, while sugar production in Indonesia is only reached 2,45 million tons per year, and the rest is imported from other countries. It does make Indonesia as the second largest sugar importer country in the world, so efforts were done to reduce the amount of imported sugar by driving sugar production. Although the productivity of sugar in Indonesia was increased, but it still does not meet the need for sugar in Indonesia because demand continues to increase while the efficiency of sugar production system is still low.

Nowadays the production of fructose syrup or High Fructose Syrup (HFS) was done as one of the essential use of sugar modification using amylase enzyme on glucoside bond breaking and isomerase enzyme in glucose units. One of the alternative materials to make fructose syrup is the sago starch. Sago acreage in Indonesia itself is the world's largest areal in the amount of 1,128 million ha or 51,3% of the world sago acreage, but the utilization of sago starch in Indonesia is still very low. In general, sago starch is only medium managed by sago farmers, so that the use of sago starch in manufacturing as fructose syrup can add value in use of sago and can replace sugar in food and beverage industry.

Kolusheva and Marinova (2006) performed an optimization of starch hydrolysis condition using α-amylase enzyme isolated from Bacillus subtilis xk-86 at various pH, temperature, and concentration of enzymes. The optimum conditions found were pH 7, temperature of 90oC, and enzyme concentration of 12 units per ml of suspension with 4,25 hours of hydrolysis time **[1]**. Rocha G, et al (2005) made comparison of the products of corn starch and dextrin in cassava. Operating conditions performed on starch concentration 30% w/w, 80 ppm $CaCl_2$, pH 6.5, 40 µL enzyme thermamyl, and temperature of 100^{o}C. Corn starch that is contained starch and amylose concentration took longer gelatinization rate time and gelatinization temperature is higher than cassava starch **[2]**. Pontoh J, and N Low (1995) perform optimization of glucose syrup manufacturing from cassava starch. Optimum conditions of liquefication stage were the concentration of starch was 15% w/v, pH 5-7, α-amylase enzyme 35 µL, temperature 80-95^{o}C for 90 minutes. Optimum conditions of saccharification stage are pH 4-5, α-amylase enzyme 60 µL, and temperature of 60^{o}C for 72 hours **[3]**. Johnson R, et al (2009) made comparison of the products of glucose and fructose in cassava and cassava starch. Concentration of Liquozyme Supra enzymes for starch cassava was 0,025% v/w when starch 0,02% v/w and the optimal operating condition 90oC, pH 6.5, hydrolysis time of 1 hour, and substrate concentration of 50% w/v **[4]**.

The purpose of this study is to learn the characteristics of sago starch and to discover the best condition of liquefication. It is to improve the utilization of sago as high fructose syrup that can be used widely in food

industry. This observational study was conducted on the characteristics of sago starch which included analysis of carbohydrate, analysis of amylose rate, moisture analysis, the determination of the temperature gelatinization, swelling and amylose leaching, and carbohydrate leaching. Observations were made on liquefication stage process in the high fructose syrup manufacturing. Liquefication stage is the stage of α 1,4-glucoside bonds fragmentation using the enzyme α-amylase to produce shorter chains of glucose called dextrin. At the liquefication stage, the observations were made on the influence of sago starch concentration and α-amylase enzyme concentration used in the acquisition of dextrin.

2. Material and Methods

Raw materials to be used was sago starch obtained from PT Bina Sagu Lestari, while the process material used was the α-amylase enzyme produced by Novo with Liquozyme Supra trademark. In this study also used CaCl2 as a supporting material, which is an enzyme cofactor of Liquozyme Supra, 0,1 M NaOH used to raise pH, and 0,1 M HCl used to lower pH. Methodological characteristics of sago starch consisting of carbohydrate analysis, analysis of the amylose, and analysis of mositure are based on the reference Standard Nasional Indonesia (SNI). The gelatinization temperature determination is done by measuring the starch suspension viscosity at different temperatures using a rheometer.

Swelling and amylose leaching properties method is based on Epriliati (2000). Starch was dissolved in water with a concentration of 2%, 6%, and 10% and heated to 55, 75, and 90^{o}C. Suspension is centrifuged at 3000 rpm for 15 minutes. Soluble solids content were observed by refractometer. Precipitate obtained was dried at a temperature of 105oC to constant weight. Swelling power were calculated by the formula: **[5]**

$$\text{Swelling Power} = \left(\frac{\text{wet precipitate weight}}{\text{dry precipitate weight}}\right)$$

Carbohydrate leaching method is based on Dubois (1956). Twenty mg of starch dissolved in 6,25mL water and heated by constant temperature silicon oil at 90, 100 and 110^{o}C for 1 hour. Suspension was centrifuged at 3500 rpm for 10 minutes. Centrifuge of 1 mL was added 1 mL of 5% phenol and 5 mL of concentrated strong acid rapidly. Mixture was stirred for 10 minutes in a water bath at 10-20^{o}C. Solution absorbance was measured at a wavelength of 520 nm with aquades as blanko solution. The amount carbohydrate leaching is determined by a standard curve **[6]**.

Reference of liquefication study method was based on Johnson R, et al (2009), Kolusheva and Marinova (2006), Rocha G, et al (2005), and Potoh J and Low N (1995) with a different variation of condition. The liquefication process was begun by making sago starch suspension to achieve the desired concentration of dry extract with 250 ml total volume. Sago starch was added $CaCl_2$ at 20 ppm and pH was set at 5,3 to 5,6 range. Starch suspension was stirred and heated until the temperature reached 90°C and added to the suspension with the variations that had been determined. Concentrations of sago starch used were 10% w/v, 15% w/v, 20% w/v, and 25 % w/v dry extract with α-amylase enzyme concentrations were 0,06% v/w, and 0,1% v/w. Samples were collected every 15 minutes until the obtained value of Dextrose Equivalent (DE) over 25 and provided the greatest yield. Enzyme inactivation was done on sample by lowering the temperature and pH. Samples were dried at a temperature of 70°C for ± 24 hours and measured DE values them using Lane Eynon titration method.

3. Result and Discussion.

Carbohydrate, amylose, water content and gelatinization temperature of sago starch can be seen in Table 1, and swelling properties is showed in Figure 1.

Table 1. Composition of Sago Starch

	Nilai
Carbohydrate	84,78 %
Amylosa	25,65 %
Moisture	14,1-17,2%
Gelatination Temperature	76-86 °C

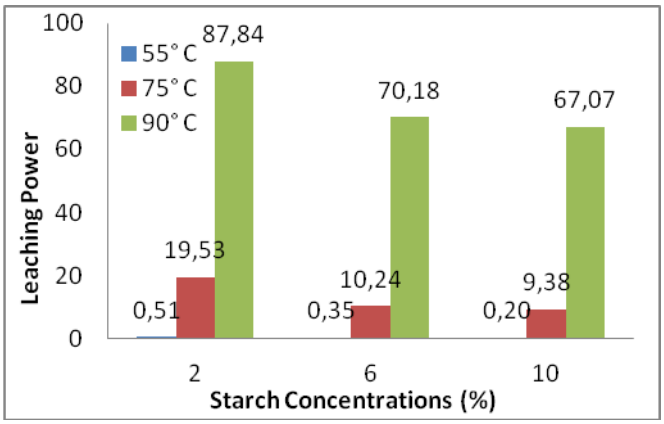

Figure 1. Swelling Properties

Based on the data of observation results shown in Figure 1, indicated higher starch concentration, then the swelling power and the amylose leaching are lower. It is happened due to the higher concentration of sago starch, kinetic energy produced by water molecules is lower because the molecular mobility of water is narrow, so that the number of water molecules into the growing molecule is low. In addition, the higher the temperature, the higher power and solubility of amylose. This is also related to the kinetic energy that is larger by the increasing in temperature. Swelling properties of sago starch shown in Figure 1 is quite high. This concern with the water absorption capacity of sago starch is high due to its swelling power associated with water absorption and gel formation. This condition indicates sago starch processing does not require too much energy. A high swelling power is also caused by high rate of amylose because amylose fraction of starch is soluble in water. This is due to amylose has a weak hydroxyl group **[6]**.

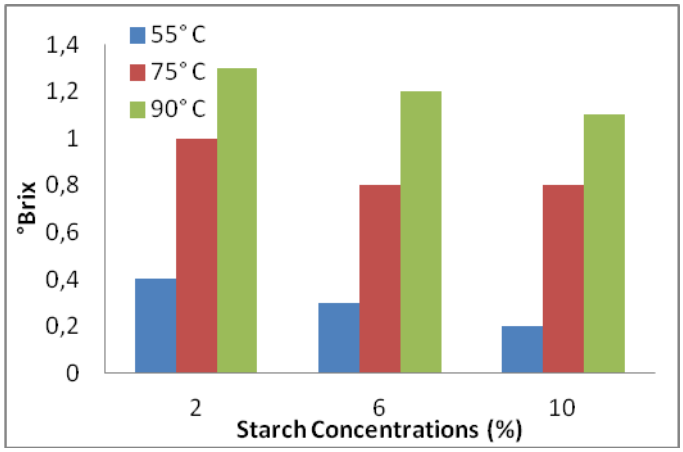

Figure 2. Amylose Leaching Properties

Amylose leaching properties is shown in Figure 2. Figure 2 shows the higher the concentration and temperature, the lower the degree of amylose leaching. At gelatinization, amylose is released from granule. Thus, when gelatinization is occurred, the released amylose fraction is high so the degree of solubility is higher.

Carbohydrate leaching is shown in Figure 3. Figure 3 shows the heating temperature influences the efficiency of gelatinization process. It can be seen that at the uniform time of heating, the higher the temperature, the higher the concentration of leaching carbohydrates. It is also related to the kinetic energy of water along the greater the temperature increasing. The higher the temperature, the starch molecules are more soluble. The result of this observation can be considered for the determination of the heating temperature so as to streamline the time and energy for starch gelatinization process.

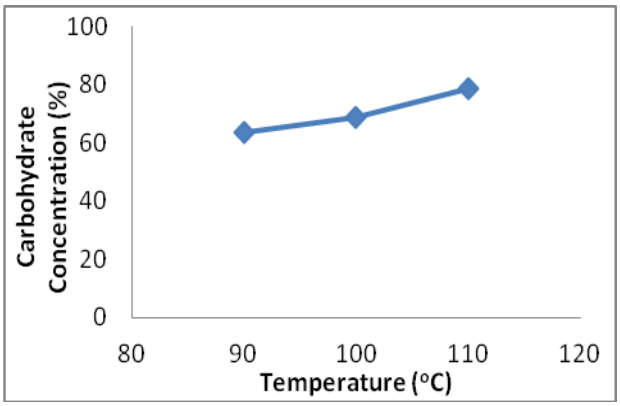

Figure 3. Carbohydrate Leaching

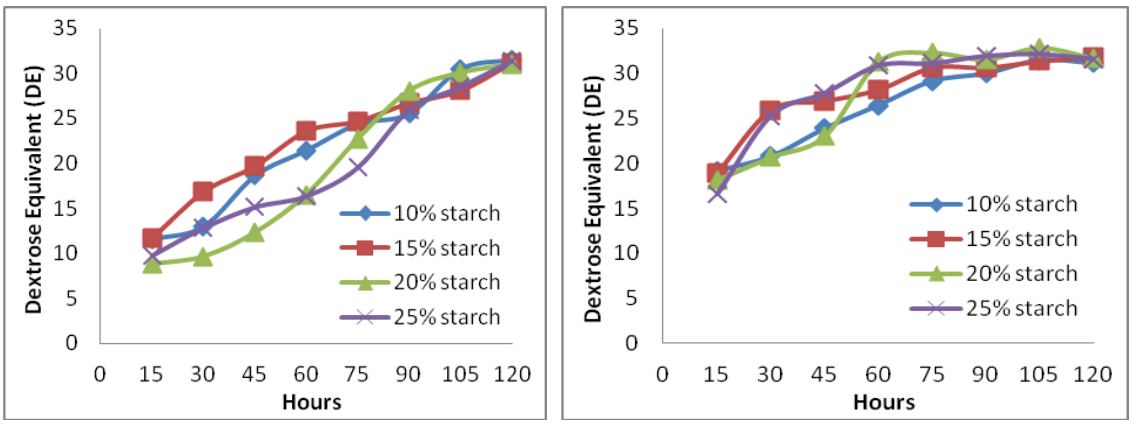

Figure 4. Dextrose Equivalent Conversion Profile at Enzyme Concentration of 0,06% (left) and at Enzyme Consentration of 0,1% (right)

The results of liquefication stage can be seen in Figure 4. In Figure 4, can be seen that the longer the hydrolysis time, the greater the value of DE that is obtained. It is due to the longer hydrolysis time, the more starch to have contact with enzymes that cut more polysaccharide chains. The purpose in this study is to find the enzyme concentration and the concentration of starch hydrolysis in order to obtain the shortest possible time with DE values above 25. Based on the Figure 4, the time needed to obtain dextrin with DE values above 25 for the enzyme concentration was 0,06% at 90 minutes, whereas for 0,1% concentration, it took a shorter time which is at around 30-75 minutes. It shows that the greater the concentration of enzyme used in the hydrolysis, the shorter time is needed, since the amount of enzyme that will cut the polysaccharide chains become more. At 0,1% enzyme concentration, hydrolysis time of 30 minutes in the fastest in starch concentration 15% and 25%. So further consideration is needed in determining the concentration of starch to be used for saccharification stage, it is done by calculating the revenue dextrin formed. Dextrin yield formed can be seen in Table 2.

Tabel 2. Dextrin Yield at Enzyme Concentration 0,1%

Enzyme Concentration	Starch Concentration	
	15% w/v	25% w/v
0,1% v/w	20,45%	64,20%

From Table 2, it can be seen that the higher starch concentration, the more starch is converted into dextrin. It was caused by the increasing number of starch that can be broken down into dextrin, so that the resulting yield is becoming increasingly large. So it can be concluded that the concentration of starch can provide the highest dextrin is 25%. At this liquefication stage, is concluded that the concentration of starch is 25% w/v and enzyme concentration is 0,1% v/w can provide the fastest time is 30 minutes with the greatest yield of dextrin. Then the optimum condition of liquefication stage is conducted by trials at this stage. Saccharification stage level is the stage of further fragmentation of glycoside chain using the enzyme glucoamylase enzyme to produce simple glucose cluster with DE values ranging from 94-98. Dextrose equivalent conversion profile at saccharification stage in time of 30 minutes can be seen in Figure 5.

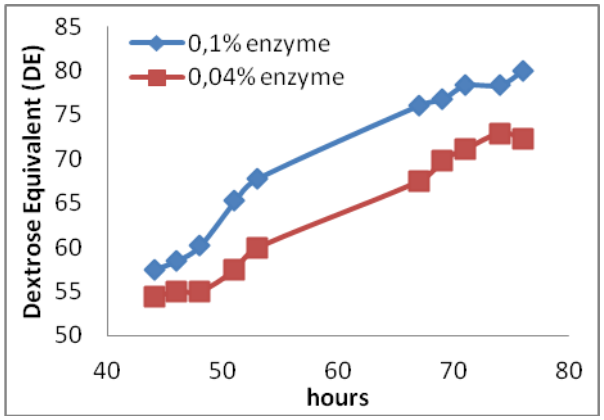

Figure 5. Dextrose Equivalent Conversion Profile at Saccharification Stage in Liquefication Time of 30 Minutes.

In Figure 5, can be seen that both the enzyme concentration 0.04% and 0,1%, did not produce glucose solution with DE values above 94. It is possible due to the solution viscosity dextrin which is thickened due to the short liquefication process, so that glucoamylase enzyme performance to be slightly inhibited. This case is supported by the shaker using that does not allow a homogeneous mixing of the solution with a viscosity sufficiently high. The using of shaker device as a mixer device based on this study by the hydrolysis of the length of time that must be followed, so as not to allow magnetic stirrer using. So the further study is made on the selection of the optimal variation in liquefication stage by calculating the dextrin yield at hydrolysis time of 2 hours as seen in Figure 6. It is based on the literature that the liquefication run for 2 hours.

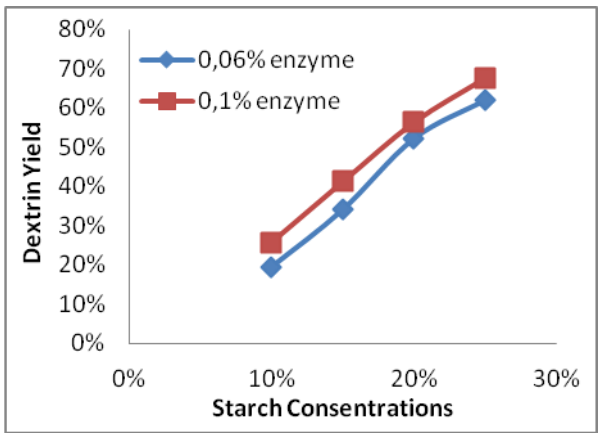

Figure 6. Dextrin Yield at Saccharification Stage in Time of 2 Hours.

In Figure 6, it can be seen that the concentration of the enzyme causes an increasing of yield. It is due to the more increasing concentration of enzyme and starch, the more amylose in starch that have contact and fragmented by the enzyme. Considering the yield obtained dextrin, so enzyme concentration is selected 0.1%, with 25% starch concentration and hydrolysis time on the liquefication 2 hours. Hydrolysis process for two hours is also

intended to achieve the high DE value, so the time required for the saccharification is shorter so as to streamline the time and energy. In the repeat study obtained saccharification with DE profile as seen in Figure 7.

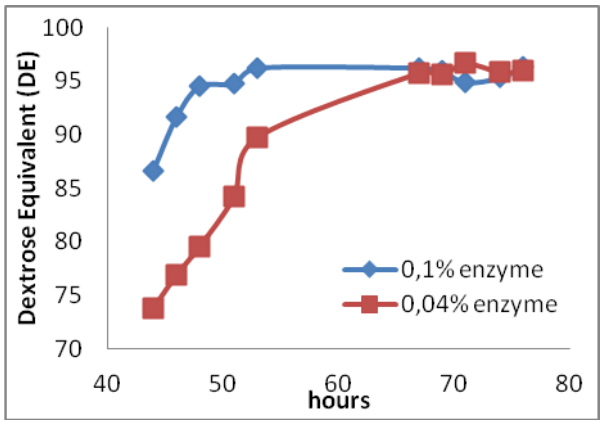

Figure 7. Dextrose Equivalent Conversion Profile at Saccharification Stage in Liquefication Time of 2 Hours.

In Figure 7 can be seen that the liquefication time of 2 hours obtained DE values of glucose syrup is above 94. Thus it can be concluded that the optimum level of liquefication conditions on the making of high fructose syrup is 25% w/v starch concentration, 0,1% v/w enzyme consentration, and 2 hours liquefication time.

4. Conclusion

Based on study results, to achieve the shortest gelatinization time, so the higher the concentration of starch used, the higher the heating temperature should be given by considering the energy used. In the liquefication stage, the best starch concentration was 25% w/v, the best enzyme concentration was 0,1% v/w with hydrolysis time of 2 hours.

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