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Modification of Sago Starch Through Lintnerization and Its Impact on Biodegradable Straw Properties

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ABSTRACT. The increasing environmental burden caused by plastic waste necessitates the exploration of sustainable alternatives such as starch-based biodegradable products. Sago starch, a renewable polysaccharide offers potential for such applications. However, its inherent limitations such as poor thermal stability and water resistance restrict its use in biodegradable product. This study investigates the effect of lintnerization, a mild acid hydrolysis process, on the physicochemical properties of sago starch and evaluates its performance in biodegradable straw production. Sago starch was modified through a 120-hour lintnerization process using 2.2 N HCl, followed by washing and drying to obtain crystalline starch. The resulting starch was characterized by XRD and SEM, and its application in straws was assessed via yield, organoleptic tests, tensile strength, water resistance, and biodegradability. The yield of crystalline starch reached 99.89%, with organoleptic scores indicating good visual and textural quality. XRD analysis revealed an increase in crystallinity from 33.32% to 49.98%, while SEM confirmed significant granule degradation. Incorporating crystalline starch improved straw water resistance up to 83.65% and tensile strength up to 7.05 MPa fulfilling industry standards. However, the biodegradability test results displayed variability due to environmental factors. These findings demonstrate that lintnerization enhances starch performance for biodegradable applications.

1. INTRODUCTION

The escalating environmental concerns associated with plastic pollution have intensified the pursuit of sustainable alternatives to conventional plastic products. Starch is a polysaccharide that is found in abundance in nature and has been identified as a highly desirable biomaterial for the synthesis of biodegradable product [1]. Sago starch extracted from the sago palm is abundant in Southeast Asia including Indonesia. Sago starch possesses inherent biodegradability, making it potential as the raw material for eco-friendly products such as biodegradable straws. However, sago starch exhibits limitations such as low thermal stability, moisture sensitivity, water absorption rate [1], high viscosity, and ease of gelatinization [2], which restrict its direct application in biodegradable products. To overcome these challenges, various modification techniques have been explored to enhance the functional properties of sago starch, one such method is lintnerization.

Lintnerization is a mild acid hydrolysis process that selectively degrades the amorphous regions of starch granules, leading to increased crystallinity and physicochemical properties. Typically, this process involves suspending starch in dilute mineral acid (HCl or H_2SO_4) at controlled temperatures (30–50°C) for extended periods ranging from days to weeks, depending on the desired hydrolysis degree. This process can produce resistant starch type III characterized by its compact and rigid structure, reduced swelling power, and enhanced thermal stability [3]. Resistant starch can be classified into 4 main types based on its physical form and origin [4]. Type I is physically inaccessible in a cellular matrix such as whole grain. Type II consist of native, crystalline starch granules while type III is retrogated starch. Type IV is a result of the chemical modification of starch. Reference [4] has investigated that this method produces 16% resistant banana starch.

Previous research by Sutoyo et al. [5] established a protocol for fabricating biodegradable straws utilizing Oil Palm Empty Fruit Bunch (OPEFB) pulp reinforced with crystalline starch and chitosan. While that study primarily optimized the cellulosic fiber composition, the results underscored the governing role of starch crystallinity in determining the product's water resistance. This study aims to address this gap by examining the impact of lintnerization on the properties of sago starch and evaluating its suitability for biodegradable straw properties, targeting the quality standards set by SNI 3451:2011 for tapioca flour as a baseline for starch quality and JIS for straw mechanics. This research was conducted to studying the development of starch-based alternatives to plastic straws. Analysis of Variance (ANOVA) is employed to determine the significance of the effects of modified starch composition on bioplastic product especially sustainable straw alternatives.

1.1 Research Objectives

The specific objectives of this research are:

1. To investigate the effect of acid hydrolysis (lintnerization) on the physicochemical properties of sago starch.
2. To characterize the morphology and crystallinity of the modified starch.
3. To formulate biodegradable straws using the modified starch and evaluate their mechanical strength, water resistance, and biodegradability.

2. MATERIALS AND METHODS

2.1 Materials

The materials used in this research include sago starch (Papua JAVARA), hydrochloric acid 37% (Merck), ethanol 96% (Nusa Kimia), Sodium Hydroxide (Merck), and aquadest.

2.2 Equipment

The equipment used in this research included beaker glasses, measuring flask, volume pipette, erlenmeyer, pH meter, basin, filter cloth, blender, oven, and Scanning Electron Microscope (SEM).

2.3 Methods

The process of modifying starch using the acid hydrolysis method (lintnerization) produce crystalline starch that resist enzymatic hydrolysis [6]. 250 grams of sago starch was suspended in 500 mL of 2.2 N HCl solution (1:2 ratio). The sago starch suspension was allowed to stand at room temperature for 120 hours. After that, the 500 mL 2.2 N HCl solution was discarded and the hydrolyzed sago starch precipitate was washed using 1 M NaOH solution until neutral. 1 M NaOH solution was discarded and the hydrolyzed sago starch precipitate was washed with 500 mL ethanol. Then filtered with a filter cloth to separate the remaining ethanol and hydrolyzed sago starch precipitate. Next, the hydrolyzed sago starch precipitate was oven dried at 50°C for 24 hours. Modification of sago starch with lintnerization process produced crystalline starch. The dried crystalline starch was in the form of rough chunks, so it was pulverized using a blender to form a fine powder. Crystalline starch is used as a raw material fo making biodegradable straw.

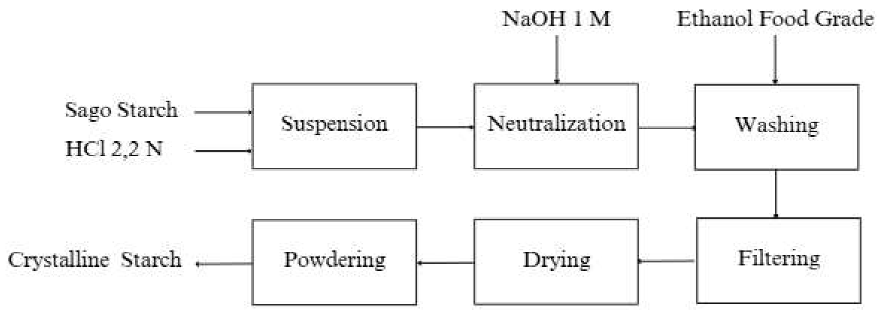

Figure 1. Process of Modifying Sago Starch

The biodegradable straws were fabricated using the thermo-pressing method established in our previous work [5]. The modified sago starch was mixed with aquadest and glycerol then heated under constant stirring until homogenized. The mixture was then poured into an iron mold and subjected to thermo-pressing at 120°C for 60 minutes to form the final straw shape.

2.4 Yield

The yield calculation determines the efficiency of the starch modification process. By calculating the yield, one may ascertain the process success rate, which is represented by the percentage difference between the end weight (W1) and the starting weight (W0) (see Eq. 1)

$$\text{Yield (\%)} = \frac{W_1}{W_0} x\ 100\% \tag{1}$$

2.5 Organoleptic Test

The organoleptic test assessed the quality of the crystalline starch based on sensory characteristics, specifically texture, odor, and color. The test involved 10 respondents who provided a sensory assessment using a hedonic scale (1–5).

2.6 Characterization of Modified Sago Starch

X-ray diffraction (XRD) used to determine the relative crystallinity of the modified sago starch. The relative crystallinity was calculated as the ratio of the crystalline area to the amorphous region from the X-ray diffractogram. The sago starch sample underwent X-ray diffraction (XRD) analysis and scanning in 2θ with range from 10°-37°. The measurement parameters included a nickel-filtered Cu-Kα radiation source (λ = 1.54 Å), a step size of 0.02° voltage of 45 kV, a current of 200mA [7]. Morphology was analyzed using Scanning Electron Microscope (SEM) at magnifications of 100x, 500x, and 1000x.

3. RESULTS AND DISCUSSION

The results of this study explore the effects of lintnerization on sago starch and its application in biodegradable straw production. These structural changes are discussed in relation to the physical performance of the biodegradable straws.

3.1 Yield Analysis

The yield is calculated from the percentage difference between the final weight (W1) and the initial weight (W0) of the starch modification process. The modified sago starch exhibited a final weight of 249.72 grams, resulting in a yield of 99.89%. The yield of the modified sago starch was influenced by the duration of the sedimentation process. The sedimentation duration played a crucial role, as longer periods promoted better separation from residual liquid, thus minimizing product loss.

3.2 Organoleptic Test Analysis

Organoleptic test aims to identify the parameters of crystalline starch, such as color, texture, and odor. This test was conducted on 10 respondents to assess the quality of crystalline starch products with sensory assessment based on physiological reactions in the form of shape, smell, and color of crystalline starch. The assessment score for the color parameter is score 1 is dark yellow, score 2 is light yellow, score 3 is slightly yellowish, score 4 is cloudy white, score 5 is pure white.

Table 1. Organoleptic Test Results of Crystalline Starch to 10 Respondents

Respondents	Color	Texture	Odor
R1	4	4	5
R2	5	5	5
R3	5	4	5
R4	4	4	5
R5	5	5	5
R6	5	4	5
R7	5	5	5
R8	4	5	5
R9	5	5	5
R10	5	5	5

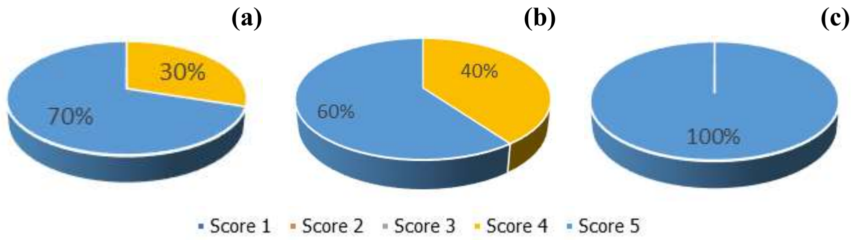

Figure 2. Distribution Diagram of Organoleptic Test Score on Color (a), Texture (b), and Odor (c) of Crystalline Starch

The respondent's results showed that for the parameters of color, texture, and odor, the dominant score was 5. That indicating pure white color, very smooth and soft texture without lumps, and no detectable odor. The tests conducted met the requirements of SNI 3451:2011 regarding Tapioca Flour Quality Standards.

3.3 X-Ray Diffraction (XRD) Test Analysis

XRD test was conducted to analyze the relative crystallinity of sago starch and crystalline starch. The analysis of the degree of crystallinity was carried out to compare the degree of crystallinity of the two types of starch which aims to determine the success of the starch modification process with the lintnerization method.

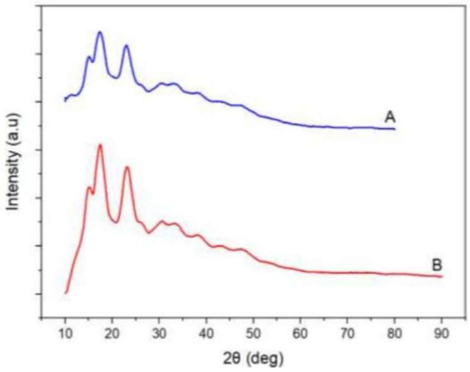

Figure 3. XRD of Sago Starch (A) and Crystalline Starch (B)

Table 2. Changes in Crystallinity and Peak Position due to Litnerization

Lintnerization Time	Crystallinity Relative	Peak Position at 2θ
0 hour (Sago Starch)	33.32%	15.09º; 17.32º; 22.97º
120 hours	49.98%	15.09º; 17.44º; 23.14º

From the XRD analysis, it can be seen that the relative crystallinity has increased. Sago starch has a relative crystallinity of 33.32%, then the modification process with the lintnerization method produces crystalline starch which has a relative crystallinity of 49.98%. This is because the amorphous part has been destroyed during the process so that it leaves the crystalline part of the starch which causes an increase in the relative crystallinity percent. This is in accordance with the research [8] that the acid modification process with the lintnerization method causes degradation by acids that can increase crystallinity. Based on the results of XRD analysis, the starch modification process with lintnerization method that produces crystalline starch is considered successful.

3.4 Scanning Electron Microscope (SEM) Test Analysis

SEM test on modified sago starch is used to determine the surface structure of crystalline starch. The following is the result of SEM test with magnification of 100X, 250X, 500X, and 1000X (b, c, d) compared to the native sago starch morphology (a) reported by [9].

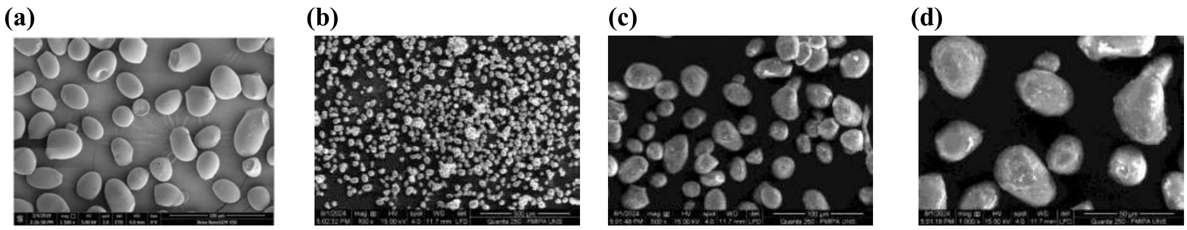

Figure 4. Morphological Analysis of Sago Starch (a) and Modified Sago Starch Using SEM (b) 100X; (c) 500X; (d) 1000X

The SEM test results of the modified sago starch showed significant changes, characterized by the fragmentation of starch granules and visible surface shrinkage. These changes indicate hydrolysis and degradation of the amorphous regions, leading to increased crystallinity. This is due to the amorphous part that has been destroyed during the process of hydrolysis process. When compared to the native sago starch morphology reported by [9], the granules appeared smooth, round, and intact with a relatively uniform size distribution and minimal surface irregularities in contrast with the modified sago starch that displayed disrupted granule structures with more compact and angular features. The results of this experiment are consistent with the research that the acid modification process with the lintnerization method causes degradation by acids that can increase crystallinity [8]. This process is characterized by the fragmentation of starch granules into smaller particles and the subsequent shrinkage of the granule surface. The SEM analysis results indicate that the lintnerization process for 120 hours effectively produces crystalline starch as evidenced by the reduction in granule size, surface roughness, and granule collapse.

3.5 Effect of Crystalline Starch Concentration on Biodegradable Straw

The incorporation of crystalline sago starch obtained through lintnerization significantly influenced the functional properties of biodegradable straws. This section elaborates on the observed effects on water resistance, tensile strength, and biodegradation behavior, as summarized in Table 3.

Table 3. Effect of Crystalline Starch Concentration on the Water Resistance, Tensile Strength, and Biodegradability of Biodegradable Straws

Factor, Sago Starch (%)	Water Resistance (%)	Tensile Strength (MPa)	Biodegradation (%)
10	78.249	5.115	19.112
15	81.316	6.126	16.277
20	83.653	7.046	15.804

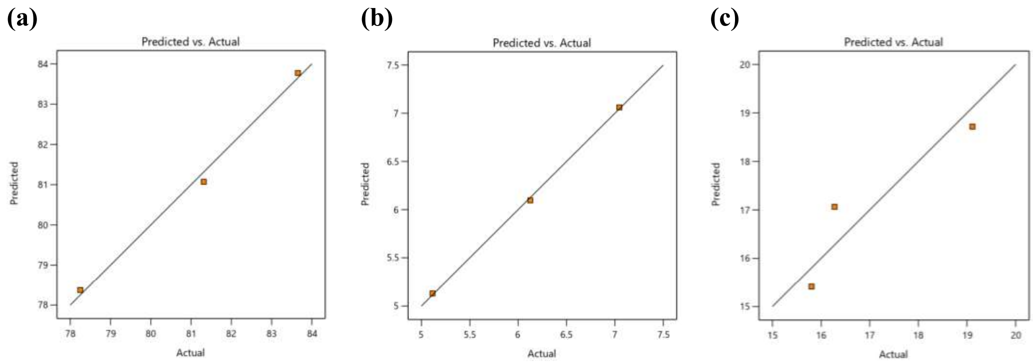

Figure 5. Water Resistance (a), Tensile Strength (b), and Biodegradation (c) of Biodegradable Straws with Different Crystalline Starch Concentrations

3.5.1 Effect on Water Resistance

Water resistance is an important component to determine the ability of biodegradable straw. The empirical data analysis revealed a statistically significant positive correlation between increasing the concentration of sago starch

(A-Sago Starch) and increasing water resistance (WR) in the formulations studied. The Analysis of Variance (ANOVA) strengthened the significance of the applied linear model (F-value = 164.40; p-value = 0.0496), which identified sago starch as a significant contributor to water resistance (WR) variability. The data and model with regard to water resistance is supported by high coefficients of determination (R^2 = 0.9949; Adjusted R^2 = 0.9919; Predicted R^2 = 0.9184). The highest water resistance value of BIOSTRAW with 20% crystalline starch was 83.653% and the lowest value was 78.249% with 10% crystalline starch. The result is congruent with with Windra et al. [10] that the modification of the starch significantly can lower the water absorption capacity, so the water resistance can be increased. According to [10], the reduction in starch's water absorption capacity following lintnerization is caused by the degradation of its amorphous domains. This structural change diminishes the quantity of available binding sites for water molecules within the granules [8].

3.5.2 Effect on Tensile strength

The highest tensile strength value of BIOSTRAW tensile strength value of BIOSTRAW with 20% crystalline starch was 7.047 MPa while the lowest value, observed with 10% crystalline starch was 5.115 MPa. Both values fulfilled Japanase Industrial Standards (JIS) which is at least 0.3 MPa. The greater the concentration of crystalline starch the tensile strength will increase. This is supported by Sutoyo et al [5]and Fadilla et al. [11] which states that the addition of modified starch can improve the mechanical properties of tensile strength. In addition, the study conducted by explained that the tensile strength value increases with degree of crystallinity. The tensile strength at 11.2% crystallinity was 5.3 MPa while at 13% crystallinity, the tensile strength reached 8.6 MPa [12].

3.5.3 Effect on Biodegradation

The results of linear regression analysis using ANOVA with the independent variable is A: sago starch and the dependent variable is B: biodegradation. The aim is to find out whether variable A significantly affects variable B. The ANOVA results show that the model is not statistically significant. The F value is 5.88, but the p value is 0.2489, which is much larger than the typical significance threshold of 0.05. This means that the variation explained by the model can easily be caused by random chance. From this result, we cannot conclude that the factor sago starch has a significant effect on response biodegradation (B). The value of predicted R^2 is -0,9610, because the value is negative, it indicating the model is performing very poorly in prediction. From the analysis, it can be concluded that the regression model for response biodegradation (B) is not statiscally significant. The variable sago starch has no proven effect on response B based on this model. The model shows very poor predictive ability (negative R^2 prediction value), and should not be used for decision making or forecasting in its current form. Biodegradation testing often gives inaccurate or incomplete result due to several environmental factors during testing. Differences in microbial community structure across soil types significantly impact the degradation rate of biodegradable materials, with higher microbial diversity generally leading to more efficient biodegradation [13]. And it also supported by environmental temperature because the higher temperatures can increase microbial activity and decomposition rates [14]. Environmental factors such as climate and soil origin also shape bacterial community patterns, which further contributes to variability in test outcomes [15]. Soil water content has also been shown to have a significant impact. Studies have shown that mineralization of biodegradable materials decreases significantly at low water content, and increases exponentially as water content increases [16]. Since the model results shows very poor predictive ability (negative R^2 prediction value), the biodegradation test results should not be used for decision making. The model requires refinement, for example by adding more test variables and considering environmental conditions that may affect the test process.

4. CONCLUSION

This study demonstrates that lintnerization effectively modifies sago starch into a more crystalline form with improved physicochemical characteristics. The 120-hour acid hydrolysis process significantly increased starch crystallinity, as evidenced by XRD analysis, and altered granule morphology, as observed in SEM imaging. The high yield (99.89%) and favorable organoleptic properties confirm the feasibility of the process. Application of crystalline starch in biodegradable straws enhanced water resistance and tensile strength, suggesting improved functional performance compared to native starch. Although the statistical model did not show a significant correlation between starch concentration and biodegradability, this is likely influenced by environmental and microbial variability during testing. Overall, lintnerized sago starch presents a promising biopolymer for developing sustainable straw alternatives, though further investigation into long-term biodegradability and model refinement is recommended.

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