Effects of Micro Powder Milling on Physicochemical Properties of Sago Starch

Abstract: In this study, we extracted sago starch from sago pith waste (SPW) using a micro powder mill. The objectives were to recover the starch from the SPW and to understand the effects of micro powder milling on the physicochemical properties of micro-powder-milled sago starch. Milling was performed at different levels of disc clearance. Native sago starch extracted from sago pith, called untreated sago starch, was used as a comparison. The results show that micro powder milling of SPW can increase the sago starch yield by around 10–17%, depending on the milling disc clearance. The amount of soluble starch in the SPW after micro powder milling was low for all treatments. The sago starch size distribution was wider at narrow clearance, but similar at wide, wide-medium, and medium-narrow clearances. Scanning electron microscopy showed that a narrow clearance treatment reduced the smoothness of the granule surface, and also reduced the starch granule birefringence. X-ray diffraction showed that the crystallinity decreased with decreasing milling clearance. Differential scanning calorimetry showed that the peak temperatures were similar at all levels, and the gelatinization enthalpy declined with decreasing crystallinity. The results suggest that sago starch can be recovered from SPW using micro powder milling, and the treatment, especially at narrow clearance, disrupts the crystalline regions of sago starch.

Key words: sago starch, Metroxylon sagu Rottb., sago pith waste, micro powder mill, physicochemical properties

INTRODUCTION

In Indonesia, sago starch is used as a staple food. One of the staple foods produced from sago starch is called "Papeda". This food is made by mixing sago starch with hot water and stirring constantly until a gel is formed. The gel has a glue-like consistency and texture, and it is eaten with fish and vegetables.

Sago (Metroxylon sagu Rottb.) starch can be obtained by wet extraction of sago pith, because sago starch is present in trunk pith. After debarking, the trunk of a mature sago palm tree contains about 250 kg of starch, 425 kg of water, and 175 kg of other materials.1) The amount of starch obtained depends to a great extent on the fineness of the grated pulp, the efficiency of washing the starch out of the grated pulp,2) and the efficiency and sophistication of the methods used.3) The mechanical process currently used to extract sago starch is inefficient and often fails to dislodge residual starch embedded in the fibrous portion of the trunk.4) Previous studies have found that the amount of sago starch trapped in sago pith waste (SPW) is around 30–45% on a dry weight basis.5) Lai et al.6) reported that the amount of sago starch in SPW varies between 58 and 67% on a dry weight basis. The SPW therefore needs to be used to reduce loss of sago starch. Several papers have been published on the use of SPW for the production of food and non-food products, such as glucose,7,8) fructose syrup,9) bioethanol,10,11) and biobuta-
The extraction of residual starch from sago waste residues using an enzyme (Pectinex Ultra SP-L) was introduced by Mohd et al.\textsuperscript{3)}

The yield of sago starch could be increased by extraction of SPW. A micro powder mill is a type of food processor that is commonly used for milling cereals and grains, and can be used to separate the sago starch physically from SPW. This method is practical, because industrial operation of a micro powder mill is easy.

Micro powder milling is a dry mechanical treatment. Dhital et al.\textsuperscript{13)} reported that mechanical treatment alters the starch granular structure, physicochemical properties, and digestibility. The physical damage caused by milling results in starch granules with irregular, rough, and less uniform surfaces, and in reduced granular crystallinity.\textsuperscript{14} Al-Rabadi et al.\textsuperscript{15)} reported that for the same grain type and grinding under similar conditions, the particle size distribution may differ as a result of variations in the mechanical properties.

The effects of mechanical treatments of starch have been investigated, e.g., the effects of cryogenic and hammer milling on rice grains,\textsuperscript{16,17} of ball milling on maize starch,\textsuperscript{18} of ultrasonication on wheat and rice starch,\textsuperscript{19} and of cryomilling on potato and maize starch.\textsuperscript{19,20} Most of these studies found that physical treatment led to changes in the crystallinity and internal granular architecture of the starch. However, there is a lack of information on the properties of sago starch produced from SPW by micro powder milling.

The objective of this study was to investigate the properties of sago starch obtained by micro powder milling of SPW. The changes in micro-powder-milled sago starch were investigated using light microscopy, scanning electron microscopy (SEM), particle size analysis, X-ray diffraction (XRD), and differential scanning calorimetry (DSC).

**MATERIALS AND METHODS**

**Materials.** Sago (Metroxylon sagu Rottb.) trunk was obtained from a sago processor in Bogor, West Java, Indonesia. To determine the yield of sago starch from sago pith, the sago trunk was cut into pieces weighing around 200 g. The pieces were suspended in 800 mL of tap water and homogenized using a blender. The homogenate was sieved through two layers of muslin cloth and allowed to settle. The precipitate was washed three times. The starch and SPW were air dried and weighed until the water content was around 10%, measured using an infrared moisture balance (FD-600-i, Kett Co., Tokyo, Japan).

The true starch yield of sago pith was measured using the following method. After separating the sago starch and SPW from the sago pith, SPW (3 g) was soaked in deionized water (120 mL) in a 200 mL Erlenmeyer flask. The sample was autoclaved at 120°C for 30 min. After cooling to 37°C, 6 U α-amylase from porcine pancreas (A3176, Sigma-Aldrich Co., St Louis, US) was added, and the sample was kept at 37°C for 24 h. After hydrolysis, the sample was filtered using a glass filter and the solution was collected. The sugar content of the solution was determined using a phenol-sulfuric acid method.\textsuperscript{21)}

True starch yield of sago pith (%) = \[ \text{Yield of starch from pith (g) + sugar content of solution (g)}/\text{sago pith (g) × 100} \] (1)

The yield of sago starch could be increased by extraction of SPW. A micro powder mill is a type of food processor that is commonly used for milling cereals and grains, and can be used to separate the sago starch physically from SPW. This method is practical, because industrial operation of a micro powder mill is easy.

Micro powder milling. The dry SPW was milled using a micro powder mill (G-008; West Co., Ltd., Niigata, Japan). The milling process was conducted using either a water coolant (WC) or ice-water coolant (IC) to reduce the heat generated by the micro powder mill. Milling was performed at different levels of disc clearance, namely wide (T1), wide-medium (T2), medium-narrow (T3), and narrow (T4). The starch and fiber were separated by decantation. The sago starch yield produced from SPW was calculated according to the following equation:

Micro-powder-milled sago starch yield (%) = Weight of sago starch (g)/weight of sago pith (g) × 100 (3)

**Scanning electron and light microscopy.** The morphologies of powder samples of T0, micro-powder-milled sago starch, and SPW samples were examined using SEM (Miniscope TM-1000, Hitachi High-Technologies Corp., Tokyo, Japan), at an accelerating voltage of 15 kV. The samples were spread thinly and evenly on circular metal stubs using double-sided adhesive tape. The birefringences of the untreated and micro-powder-milled starch were determined using polarized light microscopy (Olympus BX51, Olympus Optical Co., Ltd., Tokyo, Japan). A camera was used for image capture and micrographs were obtained at 40× magnification for each sample.

**Soluble starch analysis.** The soluble starch content of the supernatant of the micro-powder-milled SPW was analyzed in triplicate. The micro-powder-milled SPW samples (about 100 mg) were diluted with 2 mL of distilled water and then centrifuged at 1,912 × G for 10 min. An aliquot of the supernatant was collected and centrifuged at 4,302 × G for 10 min. The final supernatant was collected and the soluble starch as the total sugar content was determined, using the phenol–sulfuric acid method.\textsuperscript{21)} The apparent total sugar content was measured from a calibration curve obtained using a similarly treated glucose solution.

**Particle size analysis.** The particle size distribution of the T0 and micro-powder-milled sago starch were measured using laser diffraction (Microtrac MT3300EX2, Nikkiso Co., Ltd., Tokyo, Japan). The sample (about 5 mg) was dispersed in 25 mL of 96% ethanol solution and then ultrasonicated at 20 kHz for 20 s. From each distribution, D\(_{v,0.1}\), D\(_{v,0.5}\), and D\(_{v,0.9}\) were obtained, representing the particle diameters at cumulative volumes of 10, 50, and 90%, respectively. The size dispersion was evaluated from the dispersion indexes, using the span equation:\textsuperscript{18}i

\[
\text{Span} = (D\(_{v,0.9}\) − D\(_{v,0.1}\))/(D\(_{v,0.5}\))
\] (4)
XRD analysis. XRD was performed using an Ultima IV X-ray diffractometer (Rigaku Corp., Tokyo, Japan). T0 and micro-powder-milled sago starch (about 50 mg) were placed in a square glass cell (2 cm × 2 cm) and exposed to an X-ray beam, operated at 40 kV and 40 mA, with Cu Kα radiation (Ni filter). The 2θ scanning range was 3–33°, which covers all the significant diffraction peaks of starch crystallites. The diffraction slit was operated at 0.5–10 mm and the scan rate was 3°/min.

The method developed by Cheetham and Tao was used to calculate the percentage crystallinity of the sample from the X-ray measurements. The area above the smooth curve corresponded to the crystalline portion, and the lower area, between the smooth curve and a linear baseline, which connected the intensities at 2θ = 33° and 3°, corresponded to the amorphous section of the sample. The percentage crystallinity was calculated from the ratio of the crystalline peak area to the total crystalline area.

DSC analysis. DSC was performed using a DSC 6100 calorimeter (Seiko Instruments Inc., Chiba, Japan). Samples of T0 and micro-powder-milled sago starch (about 12 mg) were weighed into silver pans of known weight; distilled water (45 μL) was added (starch/water ratio 1:3.75 by weight), and then the silver pans were sealed hermetically. An empty sealed silver pan was used as a reference. The sample was heated from 10 to 120°C at a rate of 1°C/min. The characteristic transition temperatures, i.e., onset temperature (To), peak temperature (Tp), and conclusion temperature (Tc), and the enthalpy of gelatinization (ΔH) were determined using the built-in software. The enthalpy of gelatinization was calculated from the peak area of the endothermic curve and was expressed in joules per gram of dry starch. The experiments were performed in duplicate.

RESULTS AND DISCUSSION

Sago starch yield.
The true yield of sago starch from pith, calculated using Eq. (1), was 65% (Fig. 1). The yields of sago starch obtained using commercial methods, calculated using Eq. (2), were around 32%. The obtained results agree with those reported by Pet-Lang et al. The yields of sago starch obtained using WC and IC by micro powder milling at various disc clearances increased by around 10, 15, 17, and 18% for T1, T2, T3, and T4, respectively.

The sago starch yield at T1 was lower, because this treatment produced large particles of SPW (Fig. 2 (A)); therefore most of the starch was not released. According to Cecil and Mohd et al., the sago starch in SPW is trapped within the parenchyma cells or fibers. To liberate the sago starch, it is therefore necessary to break down the SPW. T4 treatment (Fig. 2 (D)) produced smaller SPW particles than T3 treatment did (Figs. 2 (B) and (C)); the sago starch yields were similar. Because T4 treatment produces finer sago pith, it becomes difficult to separate the starch and fibers. Cecil reported that further grating (secondary grating) gives little further increase in the yield and makes separation of the starch and fibers difficult. The soluble starch in the supernatant of micro-powder-milled SPW (Table 1) was very low, ranging from 0.13 to 0.38% and from 0.13 to 0.35% for WC and IC, respectively. This shows that micro-powder-milling treatment hardly breaks the covalent bonds in the amylose and amylopectin chains.

Starch granule shape.
The SEM micrographs of T0 and micro-powder-milled sago starch are shown in Fig. 3. The surfaces of the granules in T0 and those obtained using T1 treatment were smooth, without any pores and fissures. T0 granules are basically smooth and oval, with diameters in the range 20–40 μm, but the granule size can reach 20–60 μm. T2 and T3 treatments gave slightly rough granules, and T4 treatment resulted in loss of smoothness, with both WC and IC. This might be because friction in the T4 treatment was higher than in the other treatments. Dhital et al. reported that the level of disruption of starch granules by mechanical treatment depends on the treatment type and severity, as well as the starch source.

Images of the sago starch observed under normal light and polarized light are shown in Fig. 4. Normal light microscopy (Fig. 4 (A)) clearly shows that the sago granules were mainly oval, but some were spherical. The surfaces of T0 granules were undamaged, except in the cases of the T2, T3, and T4 treatments. When the starch was observed under polarized light (Fig. 4 (B)), T0 and starch subjected to T1, T2, and T3 treatments showed birefringence, with strong patterns at the granule centers; these typical “Maltese crosses” were uniform, with two crossed lines and a dark line at the center. However, the birefringence of the sago granules subjected to T4 treatment almost disappeared. According to Ambigaipalan et al., the birefringence patterns indicate that amylopectin crystallites are arranged radially within the granules at right angles to the surface, with their single reducing end group towards the hilum.

Particle size.
The mean diameters and spans of the sago starch obtained using different milling treatments are shown in Table 1. The

![Fig. 1. Sago starch yield obtained from sago pith (T0) and average of sago starch yields obtained from micro-powder-milled sago pith waste (SPW) at different disc clearance treatments with either water coolant (WC) or ice-water coolant (IC).](image-url)

![Fig. 2. SEM micrographs of T0 and micro-powder-milled samples with different disc clearance settings.](image-url)

![Fig. 3. Normal light microscopy images of T0 and micro-powder-milled sago starch.](image-url)

![Fig. 4. Normal light microscopy (A) and polarized light microscopy (B) images of T0 and micro-powder-milled sago starch.](image-url)
average size of T0 granules varied from 17.3 to 36.4 μm. The average sizes of all the granules of sago starch milled at T1, T2, and T3 were similar, and slightly higher than that of T0. T4 treatment gave larger granules, of average sizes from 18.0 to 108.2 μm and from 17.9 to 109.4 μm for WC and IC treatments, respectively. The $D_{v,0.5}$ value increased, indicating increasing particle size. The effect of micro powder milling on the size distribution of starch granules was not greater than that of ball milling. This might be because micro powder milling only involves friction, whereas ball milling involves friction, collisions, impingement, shearing, and other mechanical actions.28)

The span reflects the width of the starch granules. The particle size distribution index of T0 was 0.80 μm and did not differ from those obtained using wide and medium clearance (T1, T2, and T3) milling. For the T4 treatment,
Fig. 3. Scanning electron microscopy images of sago starch, untreated sago starch, and micro-powder-milled sago starch (1,000 × magnification).

(A) untreated sago starch sample (T0), (B) micro-powder-milled sample with water coolant (WC) and wide clearance setting (T1), (C) with WC and wide-medium clearance setting (T2), (D) with WC and medium-narrow clearance setting (T3); (E) with WC and narrow clearance setting (T4); (F) with ice-water coolant (IC) and wide clearance setting (T1), (G) with IC and wide-medium clearance setting (T2), (H) with IC and medium-narrow clearance setting (T3), and (I) with IC and narrow clearance setting (T4).
however, the particle size distribution was twice as broad, around 2.2 µm, with WC and IC. This might be caused by swelling of the starch granules during milling.

**XRD.**

The XRD pattern and the relative crystallinities of T0 and sago starch micro powder milled at different levels of disc clearance are presented in Fig. 5 and Table 2. As expected, T0 had a C-type diffraction pattern with peaks at 2θ values of ~15°, 17°, 18°, and 23°, and a small peak at 2θ = 5°. The relative crystallinity of T0 was around 30.5%; this is within the range reported by Srichuwong et al.\(^{29}\) and Adawiyah et al.,\(^{30}\) i.e., between 27.5% and 32.9%. The XRD patterns show that the crystallinity decreased with decreasing milling clearance, and the crystalline peaks almost disappeared for T4 treatment. The crystalline region decreased gradually from 30.5% for T0 to 11.2% and 13.3% for T4 milling treatments with WC and IC, respectively.

![Fig. 4. Birefringence changes of untreated sago starch and micro-powder-milled sago starch: (1) under normal light and (2) under polarized light at 40 × magnification; bar indicates 50 µm.](image-url)
The DSC results and gelatinization temperatures for T0 and micro-powder-milled sago starch are presented in Table 2. The onset temperature (\(T_o\)), peak temperature (\(T_p\)), and conclusion temperature (\(T_c\)) did not differ much for all treatments. The average \(T_o\), \(T_p\), and \(T_c\) values of T0 were 56.3, 66.9, and 75.9°C, respectively. For treated sago starch, the \(T_o\) values varied from 53.7 to 57.3°C, the \(T_p\) values varied from 66.3 to 66.8°C, and the \(T_c\) values varied from 71.5 to 75.9°C.

The \(\Delta H\) values for gelatinization primarily reflect the loss of molecular (double-helical) order.\(^{[23]}\) The data in Table 2 show that the enthalpy of gelatinization sharply decreased from 15.4 J/g for T0 to 3.8 J/g and 4.9 J/g for starch subjected to T4 treatment with WC and IC, respectively. The decrease in the crystalline region was related to the particle size (Table 1) and \(\Delta H\) value (Table 2). This suggests that the physical force in the T4 treatment is sufficient to disrupt the crystalline structure. The gelatinization temperature and enthalpy of the starch depend on the microstructure and degree of crystallinity within the granules, the granule size, and the amylose to amylopectin ratio.\(^{[26]}\) The results of this study showed that T4 treatment could lead to damage of the amylopectin crystalline structure, and the coolant temperature had no effect in this treatment.

Sago starch gel is firmer, and has lower adhesiveness and higher cohesiveness compared with other starches, and is suitable for food production.\(^{[32]}\) The annual demand for sago starch continues to increase, especially in Japan for use in noodle powder. However, sago starch production is limited. In this study, we managed to increase the sago starch yield from SPW by around 17% by T3 treatment. According to Ehara,\(^{[33]}\) Indonesia produces approximately 208,000 t of sago starch per year and the amount of starch discarded as SPW is around 68% (442,000 t). T3 micro powder milling for secondary recovery of SPW could increase sago starch production by around 75,140 t/year. Schemes should be sought for taking advantage of this as it is an important industrial theme for overcoming shortages of this starch.

**CONCLUSION**

The sago starch from SPW left behind after starch extrac-

![Fig. 5. X-ray diffraction patterns of untreated sago starch and micro-powder-milled sago starch.](image)

(A) Water coolant, (B) ice-water coolant. T0, untreated sago starch sample; T1, micro-powder-milled sample with wide clearance setting; T2, with wide-medium clearance setting; T3, with medium-narrow clearance setting, and T4, with narrow clearance setting.

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**Table 2.** Relative crystallinities and gelatinization properties of sago starch obtained using various micro-powder-milling treatments.\(^{[a]}\)

| Treatment  | RC (%) | \(T_o\) (°C) | \(T_p\) (°C) | \(T_c\) (°C) | Range (\(T_c - T_o\)) (°C) | \(\Delta H\) (J/g) |
|-----------|--------|-------------|-------------|-------------|---------------------------|-------------|
| T0        | 30.5 ± 1.8 | 56.3 ± 0.3 | 66.9 ± 0.0 | 75.9 ± 0.1 | 19.6 ± 0.1 | 15.4 ± 0.5 |
| WC-T1     | 30.1 ± 1.6 | 56.1 ± 1.3 | 66.5 ± 0.0 | 74.9 ± 0.1 | 18.8 ± 1.4 | 14.5 ± 0.7 |
| WC-T2     | 22.4 ± 0.7 | 57.0 ± 0.2 | 66.8 ± 0.1 | 75.8 ± 1.6 | 18.8 ± 1.4 | 12.1 ± 0.0 |
| WC-T3     | 22.1 ± 1.1 | 56.3 ± 0.3 | 66.3 ± 0.1 | 74.6 ± 0.0 | 18.3 ± 0.3 | 11.9 ± 0.6 |
| WC-T4     | 11.2 ± 1.1 | 53.7 ± 1.6 | 65.3 ± 0.5 | 71.5 ± 1.3 | 17.8 ± 0.2 | 3.8 ± 0.6 |
| IC-T1     | 29.8 ± 1.2 | 57.3 ± 0.1 | 66.7 ± 0.1 | 74.5 ± 0.2 | 17.2 ± 0.3 | 13.0 ± 0.9 |
| IC-T2     | 24.4 ± 0.9 | 56.8 ± 1.8 | 66.8 ± 0.1 | 75.1 ± 0.6 | 18.3 ± 2.4 | 13.6 ± 1.0 |
| IC-T3     | 23.2 ± 1.6 | 55.8 ± 0.6 | 66.8 ± 0.1 | 73.9 ± 0.6 | 18.1 ± 1.3 | 12.5 ± 1.2 |
| IC-T4     | 13.3 ± 0.9 | 56.7 ± 2.4 | 65.7 ± 0.1 | 73.6 ± 0.2 | 17.0 ± 2.6 | 4.9 ± 0.9 |

\(^{[a]}\)Parameters are given as average value ± standard deviation. RC, relative crystallinity; \(T_o\), onset temperature; \(T_p\), peak temperature; \(T_c\), conclusion temperature; \(\Delta H\), enthalpy.
tion can be extracted by micro powder milling. Industrial-scale micro powder milling is more practical and efficient than other milling processes because it is a continuous system and has a short milling time. Micro powder milling of SPW can increase the sago starch yield. The best result for micro powder milling of SPW was achieved by medium-narrow treatment (T3), which resulted in a 17% increase in sago starch production; the starch properties were similar to those of untreated sago starch (T0). Physical changes in the starch granules, such as changes in the starch surface, granule size, relative crystallinity, and gelatinization properties, predominantly occurred during narrow treatment (T4).

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