

Effects of Wet Milling Sago Pith Waste on Yield and Physical Properties of Sago Starch

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Abstract— The objective of this study was to investigate sago starch recovery from sago pith waste using wet milling, and to examine the sago starch physical properties. The results show that recovery of sago pith waste by wet milling increased the amount of starch by 21% (dry basis). Scanning electron microscopy showed that wet milling did not change the smoothness of the granule surface, and birefringence was clearly seen when the sample was observed using polarized light microscopy. The volume-median diameter of wet-milled sago starch was slightly higher than that of untreated sago starch; however, the particle size distribution index (span) was the same. X-ray diffraction showed that the crystallinity of wet-milled sago starch was similar to that of untreated sago starch. Differential scanning calorimetry showed that the peak temperatures and gelatinization enthalpies of untreated sago starch and sago starch milled using a super mass colloid were the same. These results show that wet milling is better than dry milling of sago starch, and does not much change its physical properties.

Index Term-- wet milling, sago pith waste, sago starch, physical properties

I. INTRODUCTION

Sago palm, *Metroxylon sagu* Rottb., is believed to have originated on the island of New Guinea and then spread to other countries in Southeast Asia [1]. Sago starch is a staple food in Papua New Guinea and the eastern part of Indonesia. Sago has recently attracted much attention because it is an excellent plant carbohydrate source, with potential food applications [2]. Sago starch can be considered as a future source of food for several reasons: approximately 60 million t per annum of sago starch are produced in Southeast Asia alone [3]; it has a high yield per land area, around three to four times those of rice, corn, and wheat [4]; and sago starch gel is firm, with low adhesiveness and high cohesiveness. Sago starch can be used to produce many types of food such as noodles [5], bread [5]; [6], and vermicelli [7], and also can be used as a

thickener in the production of soup and baby food and as an additive in various food products [8].

Sago starch accumulation in the trunk of the sago palm can reach 250 kg (dry basis) [1]. Sago starch can be obtained by wet extraction of sago pith, after a rasping process. The extraction of starch from the sago trunk involves tree felling, cutting the trunk into logs, debarking, pith crushing, starch extraction, starch filtering, and drying and packaging [1], [9]. Sago starch extraction methods can be classified as traditional or modern. The methods differ in terms of the scale of operation and the tools that are used for processing [8], [10], [11]. Several studies have shown that the yields of sago starch obtained using traditional methods are low, and the starch is low quality [2], [8]; the yield is 25–68% of the total starch in the sago trunk [9], [12], [13]. Variations in the sago starch yield are caused by the maturity of the sago palm tree, the extraction method, and the equipment used.

Approximately 56% of the global total of sago is grown in Indonesia [1], 95% of which is growth in Papua Province. However, sago starch production in Papua Province is only around 350,000 t per year or about 7% of the potential total sago starch production [14]. The traditional method is used by the majority of sago farmers in the main producing area [15]. Sago starch production could be increased by re-extraction of sago pith waste (SPW); a previous study showed that the amount of sago starch left behind in SPW varies between 58% and 67%, on a dry weight basis [16]. Unextractable starch in SPW accumulates in the parenchymal cells embedded in fibers [17], and therefore the cellulosic cell walls must be broken down to liberate the starch [4].

Sago starch production can be increased by recovering sago starch by milling SPW. Milling or grinding of agricultural products is commonly performed to break them down and reduce the size of materials. Wet milling with a super mass colloid is used to grind and extract materials continuously,

and is easy to operate in mass production. Milling of SPW with a super mass colloidizer could be used to increase the sago starch yield. The fibers are fragmented into smaller particles and the starch granules are liberated during milling. A super mass colloidizer mill can be used in continuous milling and easily used for mass production in traditional extraction methods. However, the recovery of sago starch using wet milling has not been reported. In the present study, the yield and physical properties of sago starch produced using a super mass colloidizer mill were investigated.

II. MATERIALS AND METHODS

A. Materials

Sago palm (*M. sagu* Rottb.) logs were obtained from a sago processor in Kendari, Southeast Sulawesi Province, Indonesia. Porcine pancreas α -amylase (A3176) was purchased from Sigma-Aldrich, St Louis, MO, USA. All chemicals used in the study were analytical grade.

B. Sago Starch Yield

Sago starch extraction was performed using a commercial method. The calculation of the true starch yield for the sago pith using Eq. 1; the sago starch yield of the commercial method using Eq. 2; as well as the water contents of the starch and the SPW refer to the method reported by Santoso et al. [18].

C. Sago Pith Waste Milling

Sago pith waste (SPW) was milled using a super mass colloidizer (MKZA 10-15J; Masuko Sangyo Co., Ltd., Saitama, Japan) with an MKE10-46K disk (ϕ 250 mm). The SPW (1 kg) was milled with tap water and passed through the mill three times. The starch and fiber were separated by decantation. The super-mass-colloidizer-milled (SMCM) sago starch yield produced from SPW was calculated using Eq. 3 [18].

D. Physical Properties

The physical properties of the starch were investigated using scanning electron microscopy (SEM), light microscopy, particle size analysis, X-ray diffraction (XRD), and differential scanning calorimetry (DSC), using the methods described by Santoso et al. [18]. The physical properties of untreated sago starch were examined for comparison.

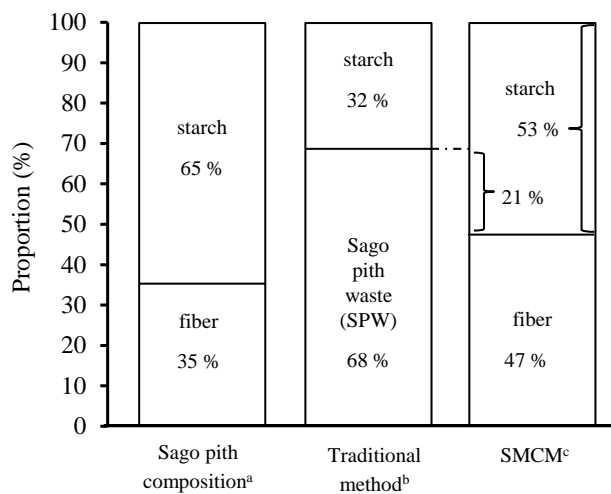
III. RESULTS AND DISCUSSION

A. Sago Starch Yield

In this study, the true starch yield of debarked sago pith was 65% (dry basis), calculated using Eq. 1 (Figure 1). However, the amount of extractable starch obtained using a commercial method, calculated using Eq. 2, was 32% (dry basis) (Figure 1), i.e., clearly lower than the true starch yield. This yield is typical of those obtained using commercial methods [8]. The amount of starch extracted varies depending on the efficiency and sophistication of the method used [12]; some methods fail

to dislodge the residual starch embedded in the fibrous portion of the trunk. SEM images show that a large amount of starch is present in the SPW, and is covered by parenchymal cells (Figure 2A).

According to Anderson et al. [19], some starch inside the polymer matrix is difficult to liberate because of microstructural cellulose embedded in the polysaccharide and protein matrix, surrounded by an outer layer of pectic material. However, some parenchymal cells were broken down and the sago starch was liberated when the SPW was subjected to wet milling with a super mass colloidizer (Figure 2B). The yield of SMCM sago starch obtained, calculated using Eq. 3 (Figure 1), was 21% (dry basis). This yield is higher than the sago starch yield obtained using dry milling, 18%, reported by Santoso et al. [18].



[a] True sago starch content in sago pith, evaluated using Eq. 1.

[b] Average sago starch yield obtained using commercial method, evaluated using Eq. 2.

[c] Average sago starch yield from SPW obtained by super mass colloidizer milling, evaluated using Eq. 3.

Fig. 1. Average sago starch yields produced from sago pith and SPW

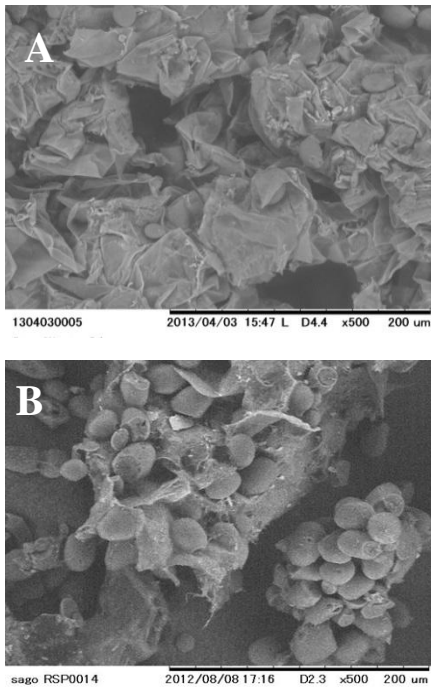


Fig. 2. SEM images of SPW (A) and SMCM SPW (B)

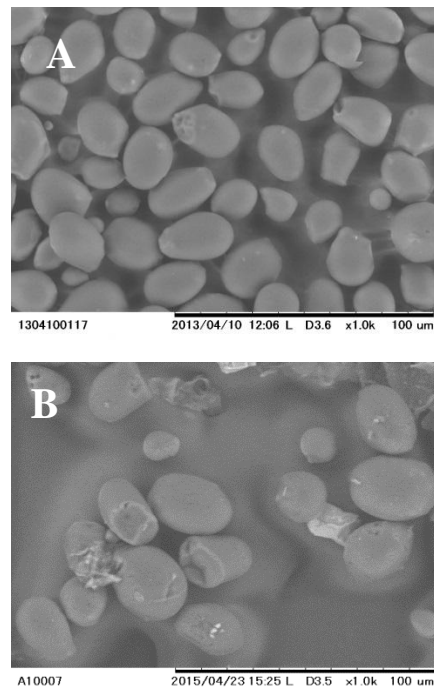


Fig. 3. SEM images of untreated sago starch (A) and SMCM sago starch (B)

B. Starch Granule Shape

SEM images of the untreated and SMCM sago starch are shown in Figure 3. The native starch granules were mainly oval, but some were spherical (Figure 3A). The surface was smooth without any fissures and pores. Fannon et al. [20] reported that although they are sometimes unobservable using SEM, all starch granules may contain pores and channels to allow passage of water, reagents, and enzymes. The granule diameter was 20–60 μm . The SMCM starch granules also had smooth surfaces, and they retained their shape and were intact (Figure 3B).

When untreated and SMCM sago starch were observed using polarized light microscopy (Figure 4), they both showed a strong dark Maltese cross at the granule center, with two crossed lines, and no loss in birefringence. 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 [21]. Birefringence and the characteristic Maltese cross generally appear in all native starch samples [22], [23]. These results indicate that the sago starch granules did not suffer major structural damage during milling, although super mass colloid mill is a mechanical treatment. However, the sago starch granule surface changed and the birefringence disappeared when sago starch recovered from SPW was subjected to dry milling using a micro-powder mill with a narrow disk clearance [18].

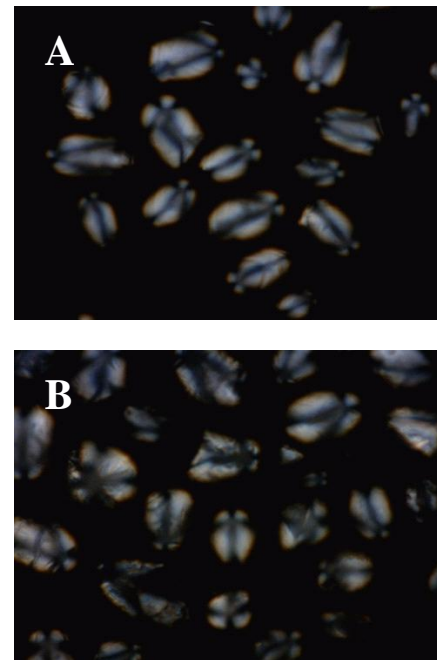


Fig. 4. Microscopy photographs of untreated sago starch (A) and SMCM sago starch under polarized light at 40 \times magnification; bar indicates 50 μm

C. Particle Size Distribution

The data in Table 1 show that $D_{(v,0.1)}$ and $D_{(v,0.9)}$, and $D_{(v,0.5)}$, representing the particle diameters at cumulative volumes of 10%, and 90%, and the median diameter, respectively, of SMCM sago starch, were slightly larger than those of untreated sago starch. The volume-median diameter, $D_{(v,0.5)}$, of

untreated sago starch was 23.90 μm , whereas that of SMCM sago starch was 30.11 μm . This confirms that the volume-median diameter increased slightly as a result of milling. However, the particle size distribution (span) of untreated sago starch was the same as that of SMCM sago starch. Recovery of sago starch from SPW by dry milling with a micro-powder mill caused swelling of the starch granules to twice the size of those of the untreated sago starch [18]. Tran et al. [24] reported that the volume-median diameter of hammer-milled rice flour particles (dry milled) was larger than that of cryogenically milled rice flour (wet milled), and, at a similar volume-median diameter, the damage to the starch granules was greater for the hammer-milled rice flour than for the cryogenically milled counterpart.

Table I
Particle size distributions of untreated and SMCM sago starch

	Untreated sago starch ^a	SMCM sago starch ^a
$D_{(v,0.1)}$ (μm)	17.31 \pm 0.16	21.34 \pm 0.19
$D_{(v,0.5)}$ ^b (μm)	23.90 \pm 0.11	30.11 \pm 0.32
$D_{(v,0.9)}$ (μm)	36.93 \pm 0.61	46.25 \pm 0.47
Span ^c	0.80 \pm 0.04	0.80 \pm 0.00

[a] Parameters are given as average value \pm standard deviation

[b] Median diameter

[c] Span: size dispersion index (Liu et al., [25])

D. XRD

The XRD pattern of untreated sago starch had strong peaks at $2\theta = 15.04, 17.06, 17.70,$ and 22.82° , and a weak peak at 26.38° (Figure 5); this is in good agreement with the results reported by Adawiyah et al. [26]. However, Lai et al. [16] and Ahmad et al. [27] observed a weak peak at $2\theta = 5^\circ$. The results show that untreated sago starch has a C-type diffraction pattern, i.e., a mixture of A-type and B-type diffraction [27], [28]. The peak positions in the XRD pattern of SMCM sago starch were similar to that of untreated sago starch. The peaks represent crystalline regions in the starch granule. XRD was also used to study the changes in starch crystallinity brought about by physical treatment, using SMCM sago starch granules. The crystallinity of the untreated sago starch was similar to that of SMCM sago starch (Table 2), suggesting that super mass colloid mill did not reduce the sago starch crystallinity. In a previous study, Santoso et al. [18] found that the crystalline regions decreased to 27.5% and 63.3% when the SPW was dry milled using a micro-powder mill at medium and narrow disk clearances, respectively.

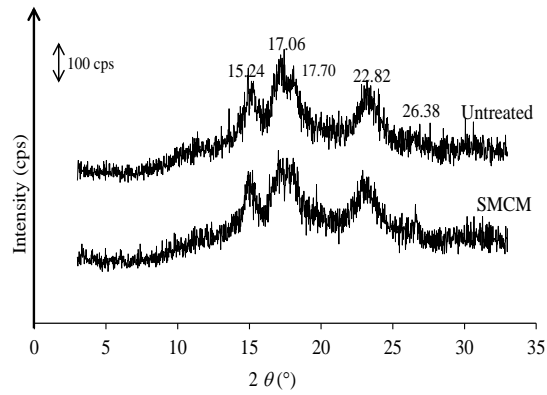


Fig. 5. XRD patterns of untreated and SMCM sago starch

E. Gelatinization Properties

The gelatinization properties of untreated and SMCM sago starch were investigated using DSC; the results are shown in Figure 6, and the gelatinization properties are summarized in Table 2. The endothermic curve and melting temperature range ($T_c - T_o$) of SMCM sago starch were similar to those of untreated sago starch. The onset temperature (T_o), peak temperature (T_p), and conclusion temperature (T_c) of SMCM sago starch were 60.5°C , 70.0°C , and 76.8°C , respectively, and did not differ from the values for untreated sago starch. The T_p values of untreated and SMCM sago starch were 66.9°C and 70.0°C , respectively. The reported values T_p of native sago starch determined using DSC vary approximately 70°C [27] and 67.3°C [26].

The gelatinization enthalpies (ΔH) of untreated and SMCM sago starch were 15.4 and 13.4 J/g, respectively (Table 2). The ΔH values are consistent with the crystallinity values obtained using XRD. ΔH of starch gelatinization is the amount of energy needed to convert the crystalline structure to an amorphous structure, and therefore reflects the degree of crystallinity of the starch granules [29], [30]. The gelatinization temperature and enthalpy of the starch depend on the microstructure and degree of crystallinity [27]. The gelatinization values suggest that super mass colloid mill did not change the microstructure and degree of crystallinity of the starch granules. Chen et al. [31] and Yeh [32] also found that wet grinding of rice produced the lowest amount of damaged starch. However, ΔH decreased sharply for recovery of sago starch by dry milling with a micro-powder mill at a narrow disk clearance [18].

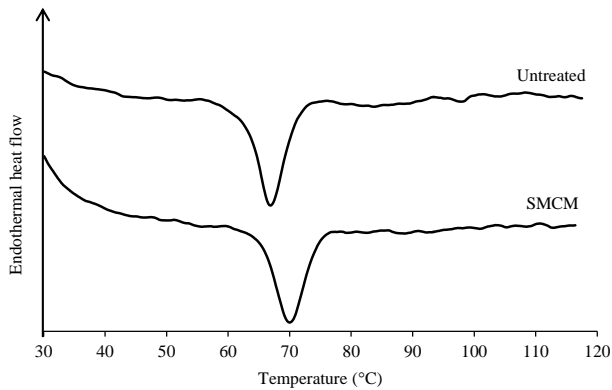


Fig. 6. DSC thermograms of untreated and SMCM sago starch

Table II
Crystallinities and gelatinization characteristics of untreated and SMCM sago starch

	Untreated sago starch ^a	SMCM sago starch ^a
RC (%)	30.5 ± 1.8	28.9 ± 1.4
Gelatinization parameters		
To (°C)	56.3 ± 0.3	60.5 ± 0.3
Tp (°C)	66.9 ± 0.0	70.0 ± 0.1
Tc (°C)	75.9 ± 0.1	76.8 ± 0.4
Range (Tc - To) (°C)	19.6 ± 0.4	16.3 ± 0.5
ΔH (J/g)	15.4 ± 0.5	13.4 ± 0.2

[a] Parameters are given as average value ± standard deviation
RC: relative crystallinity; To: onset temperature; Tp: peak temperature; Tc: conclusion temperature; ΔH: enthalpy

IV. CONCLUSIONS

Wet milling using a super mass colloidizer can be used for recovery of sago starch from SPW. The sago starch yield from debarked sago palm obtained using a commercial method was 32%, and increased by approximately 53% when SPW was recovered using a super mass colloidizer mill. The physical properties of SMCM sago starch and untreated sago starch were the same. The span value was similar for both untreated and SMCM sago starch. Milling with a super mass colloidizer did not change the XRD patterns, and the gelatinization properties of SMCM sago starch were similar to those of untreated sago starch. These results suggest that the yield and physical properties of sago starch obtained by wet milling using a super mass colloidizer were better than those achieved in a previous study by dry milling using a micro-powder mill.

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