SAGO PALM 10: 73–78 (2003)

Characteristics of Sago residue as a lignocellulosic resource I Anatomical and physicochemical properties

Sei Sasaki[1], Masaharu Ohmi[1], Hiroshi Tominaga[1], and Kiyoharu Fukuda[1]

[1] Tokyo University of Agriculture and Technology

Abstract In this study on the effective utilization of sago residue, analysis of sago residue as a lignocellulosic resource was undertaken. The cellular components and the cell sizes in deconstructed sago residue were examined. These results showed that sago residue is predominantly composed of parenchyma cells (The occupation area ratio was approximately 85%). In addition, the fiber and vessel contents were low, and these elements were remarkably damaged. These findings indicate that the sago residue is not suitable as a source of raw fiber material. The various properties of sago residue as a powder sample, which include grindability, internal surface area, and crystallinity of cellulose and starch, were then evaluated. From these analyses, it was clear that sago residue is more easily ground than Sugi (*Cryptomeria japonica*), and the crystallinity index for cellulose was reduced after simple processing. In the sago residue, the value of the internal surface area is higher than that of regenerated cellulose. Because sago residue possesses these properties, sago residue is suitable for use as a powder raw material in chemical modifications.

Key words: cellular component, crystallinity, grindability, internal surface area, sago residue

サゴヤシ澱粉抽出残渣の木質資源的性質 I 組織的・物理化学的性質

佐々木靖[1]・近江正陽[1]・冨永洋司[1]・福田清春[1]

[1] 東京農工大学農学部　〒183–8509　東京都府中市幸町 3–5–8

要約　本研究ではサゴヤシ澱粉抽出残渣(以下サゴ残渣)の有効利用を目的として，木質資源的性質の分析を行った．まず，解繊したサゴ残渣試料を用いて細胞の組成及びサイズの観察を行った．その結果，サゴ残渣はほとんど柔細胞から成ることが明らかとなった(占有面積率　約 85%)．また，繊維や道管の含量は低く，しかもそれらは著しく損傷していた．これらの結果からサゴ残渣は繊維原料としては適さないことが示された．次に粉体試料としてのサゴ残渣の性質を評価するため，粉砕性・内部表面積・セルロースとデンプンの結晶性の評価を行った．これら分析の結果，サゴ残渣は，スギと比べてはるかに粉砕しやすく，セルロースの結晶性はボールミルのような簡便な処理で低下させることが可能であり，その内部表面積は再生セルロースよりも大きいことが示された．サゴ残渣はこのような利点を持つため，利用法の一つとして化学修飾用粉体原料などに適した材料であることが示された．

キーワード　細胞の組成，結晶性，粉砕性，内部表面積，サゴ残渣

Introduction

Sago palm (*Metroxylon sagu*) is currently cultivated as a starch resource around Southeast Asia and the starch from this palm is industrially utilized. However, there are several problems facing further development of the sago starch industry. One such problem is treatment of the sago residue after starch is extracted from the sago pith. This problem can be solved by developing a method for effective utilization of the sago residue.

In order to develop new methods to utilize sago residue, the properties of sago residue must first be understood. To facilitate such understanding, vari-

ous analyses were conducted to investigate the properties of sago residue. In this report, through observation of deconstructed sago residue, the cellular components, conditions and occupation area ratio were initially analyzed. Following this, the physicochemical properties, such as grindability, crystallinity index (*CrI*) of cellulose and starch, and internal surface area (ISA) of powdered sago residue were evaluated.

Experiments

Materials

Sago pith and residue were obtained from Nistei Sago Industry, Sarawak, Malaysia. All sago samples were dried at 50°C *in vacuo* for 24 hours after extraction with an ethanol-benzene (1:2 v/v) mixture for 6 hours.

Deconstructing sago residue tissue into cells

Specimens (1 mm × 1 mm × 10 mm; radial × tangential × longitudinal) cut from sago pith and an equivalent weight of powdered sago residue were used. And then, specimens were treated with peracetic acid prepared from acetic anhydride and hydrogen peroxide for delignification. This treatment was carried out at 100°C for 5–10 minutes. Samples were observed using an optical microscope in order to determine the cell types, sizes, and the occupation area ratio (OAR). The occupation area was calculated by dividing the areas of each cell type by the total cell area.

Grinding test

Grinding tests were carried out to evaluate the ease of mechanical processing of the sago residue. A sample (2.0 g) was ground using a vibration mill (TI-100; Heikou Co. Ltd.). The ground sample was placed in the top of a sieve tower comprising 42-, 60-, 100-, and 200-mesh sieves, which were arranged in decreasing order of sample size from the top. The sieve tower was then vibrated for 30 seconds. Thereafter, the remaining sample on each sieve was weighed, and the ratio was calculated as follows:

$$R = \frac{W}{A} \times 100 \qquad (1)$$

Where R is the remaining sample ratio (%), W is the weight of remaining sample on each sieve (g), and A is total recovered sample (g). Sugi (*C. japonica*) sapwood was used as a control.

X-ray diffraction

The X-ray diffraction patterns of wiley-milled (≒ 80 mesh pass) and ball-milled (200 mesh pass) sago samples were measured. The crystallinity index of cellulose was calculated from the diffraction patterns by the Segal method (Segal et al. 1959), as follows:

$$CrI = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \qquad (2)$$

Where CrI is the crystallinity index of cellulose and starch, I_{002} is the intensity of (002) the area (2θ = about 22.8°), and I_{am} is the intensity of the background (2θ = about 18°). In addition, the CrI of starch was calculated using the peak intensity at 2θ = about 15°, which corresponds to the 3b diffraction peak of the C-type crystalline structure (Hizukuri et al. 1957).

Water vapor adsorption

In order to determine the internal surface area (ISA) of the sago sample, water vapor adsorption analysis was carried out using a BERSORP 18 (Japan Bell Co. Ltd.). Experimental conditions were as follows: sample size was controlled to about an 80 mesh pass; temperature of the thermostatic chamber was 50°C; and equilibrium time was 500 seconds. ISA was calculated from water vapor adsorption isotherm by the BET-plot method (Brunauer et al. 1938).

Results and Discussions

Cellular component

A comparison of sago pith and sago residue revealed that the cells of sago residue were more damaged than those of the sago pith. Figure 1 shows parenchyma cells from sago residue and sago pith. Figure 2 depicts other types of cells from the sago

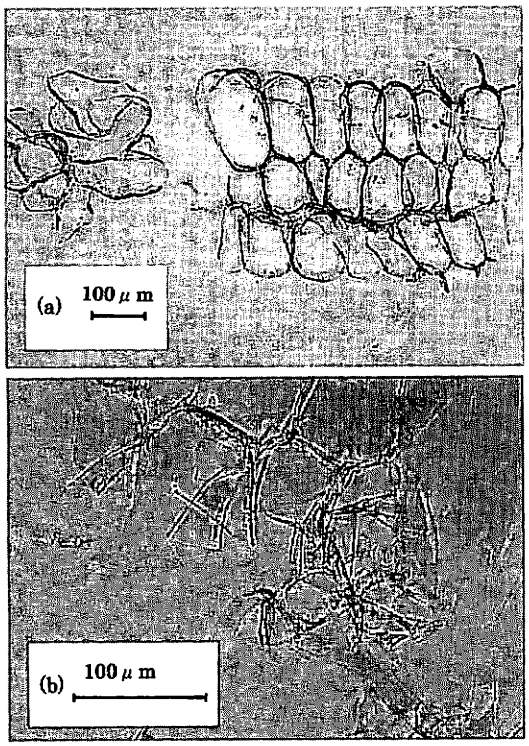

Fig. 1 Parenchyma cells observed in the pith (a) and residue (b) of sago.

palm. Figures 3 and 4 show the results of the cell size and cell wall thickness determinations, and the OAR of sago residue, respectively. These results indicated that sago residue consisted almost entirely of parenchyma cells, having an OAR of about 85%. The fiber component was damaged and accounted for a low OAR.

Sago residue was found to be unsuitable for use as a raw fiber material, because the fiber and vessel element contents were low, and those amounts that were present, which remarkably affect the mechanical strength of the products, were damaged.

Thereafter, sago residue was investigated as a raw powder material with regard to developing an effective utilization method.

Grindability

In order to evaluate the ease of mechanical processing of sago residue, the sample was ground and then the size of the particles was traced at each time. The results for sago residue are shown in Figure 5 and those for Sugi sapwood in Figure 6. The size of Sugi particles was slightly decreased for 0.5 minutes

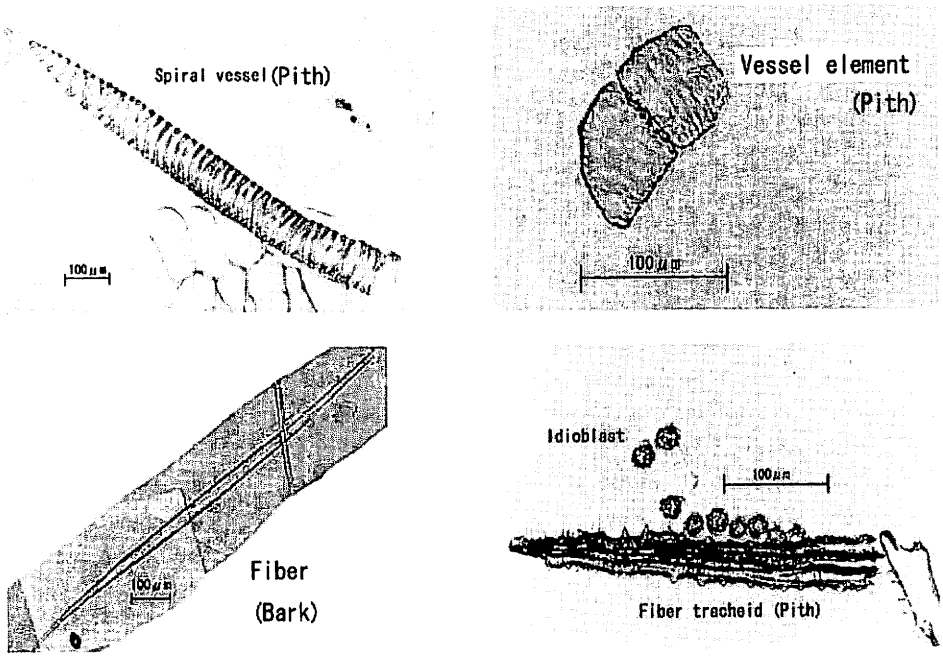

Fig. 2 Examples of each cells in sago palm.

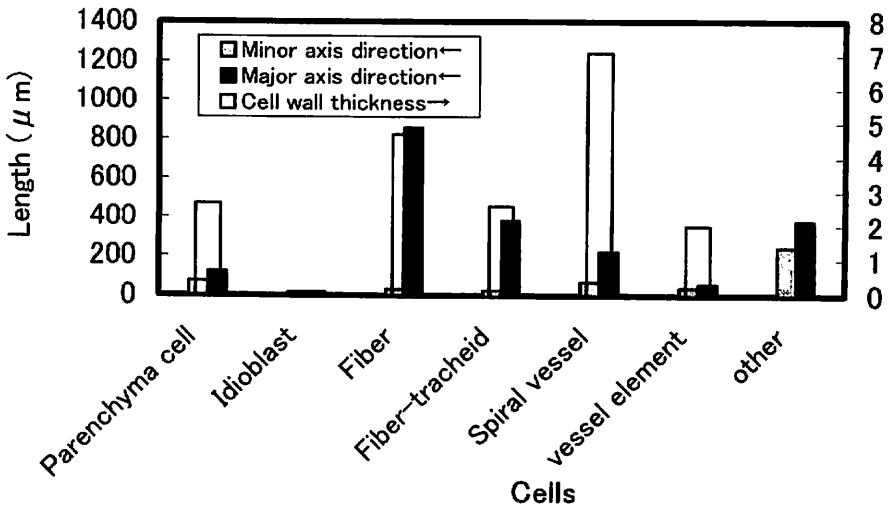

Fig. 3 The size of each cells in the sago residue.

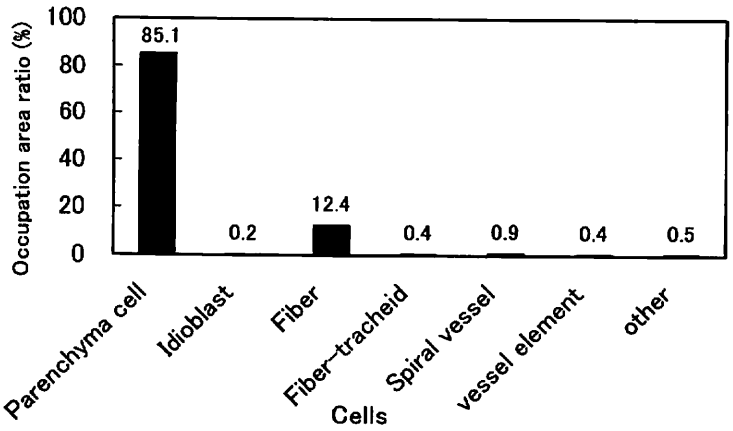

Fig. 4 OAR (%) of each cell in the sago residue.

grinding. However, the size of sago residue particles was remarkably decreased for same grinding time. Moreover, a part of the sample, which is shown as that in the "200 mesh pass" in Figure 5, passed through sieves of all mesh size in the same processing time. This result indicated that the sago residue could be easily ground. In other wards, the sago residue is a material requiring low energy in mechanical processing.

Crystallinity index (CrI)

When woody-biomass is modified to react with chemical reagents, it is first necessary to decrystallize the cellulose and starch. Chemical solvents are most often used to accomplish this, however, the preferred method is to decrystallize through mechanical processing. In this study, we investigated whether the crystallinity of cellulose and starch of sago residue was decreased by mechanical processing. The *CrI* of the cellulose and starch in sago residue was analyzed and the results obtained for rough-milled (wiley-milled) samples and fine-milled (ball-milled) samples were compared. The results are shown in Table 1, along with the literature values of the Sugi (N. Sobue et al. 1970). After ball-milling, the *CrI* of starch decreased slightly, while the *CrI* of cellulose

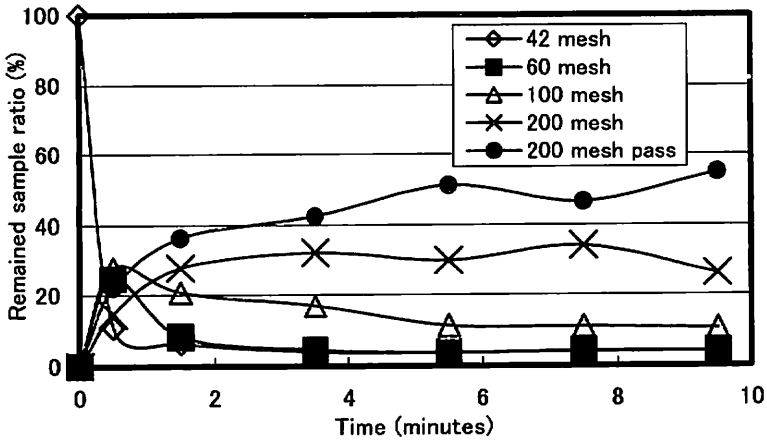

Fig. 5 Grindability of sago residue.

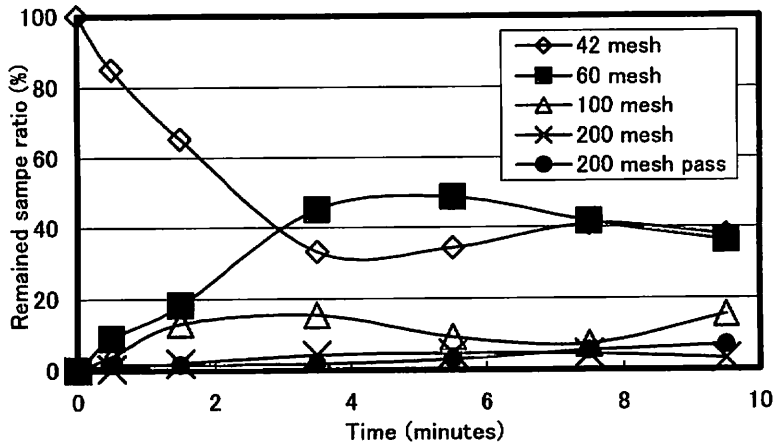

Fig. 6 Grindability of Sugi.

Table 1 Crystallinity index (%)

	Sugi[a]	Sago residue	
	(*C. japonica*)	Wiley-milling	Ball-milling
Starch	—	17	15
Cellulose	30	33	15

[a] Sobue et al. 1970

decreased remarkably. These results indicate that the crystalline regions of sago residue are easily decrystallized without addition of chemical reagents through the use of mechanical processing methods, such as ball-milling.

Internal surface area (ISA)

When sago residue was used as a raw material for chemical modification, the ISA of the sample was observed to exert a substantial influence on the chemical activity. The ISA of sago residue was therefore analyzed by water vapor adsorption, and the results are shown in Table 2. The ISA of sago residue was found to be 293 m^2/g, which is a larger value than that of regenerated cellulose (R. Yamai 1982) and other wood materials (M. Ohmi et al. 1988, S. Wang et al. 1993). In addition, the ISA of sago residue was larger than that of sago pith; the cells of the sago residue were damaged in during starch extraction, subsequently increasing the contact area with water

Table 2 Internal surface area (ISA)

	Regenerated cellulose[b]	Sugi[c] (*C. japonica*)	Hinoki[d] (*C. obtusa*)	Sago	
				residue	pith
ISA (m^2/g)	254~288	187	251	293	280

[b] R. Yamai. 1982. [c] S. Wang et al. 1993. [d] M. Ohmi et al. 1988.

vapor. These findings correspond to the observed condition of the cells, as shown in Figure 1.

Conclusions

In an effort to develop an effective method for utilizing sago residue, various properties of the residue were evaluated, and the following conclusions were obtained.

1. Sago residue consisted almost entirely of parenchyma cells. On the other hand, fibers and vessels, which influence the mechanical strength of products, were damaged, and the contents were remarkably low.

This result indicated that sago residue was unsuitable for use as a raw fiber material.

2. Sago residue had better grindability than the Sugi sapwood.

3. The crystalline region of sago residue can be readily decrystallized, without addition of chemical reagents, through mechanical processing methods, such as ball-milling.

4. The ISA of sago residue was found to be 293 m^2/g, which is larger than that of regenerated cellulose and other types of widely used wood.

Based on these results, we believe that sago residue is more suitable for use as a powder material for chemical modification than as a raw fiber material. As a raw material for chemical modification, sago residue has numerous advantages, such as a large ISA, low energy requirements for mechanical processing, and easily decreased crystallinity.

Acknowledgement

We express our gratitude to staff of the Tokyo Metropolitan Industrial Technology Research Institute for their help in the X-ray diffraction and water vapor adsorption analyses. This work was supported by a Sasakawa Scientific Research Grant from The Japan Science Society.

References

Brunauer, S., P.H. Emett, and E. Teller 1938 Adsorption of Gases in Multimolecular Layers. Journal of American Chemical Society. 60: 309–316.

Hizukuri, S. and Z. Nikuni 1957 X-Ray Diffractometric Studies on Starches. II. Structure of "C"-Type Crystallite. Nougeikagakugakkaishi. 31 (7): 525–527 (in Japanese)

Ohmi, M. and M. Suzuki 1988 Water-vapor adsorption character of woodmeal and calcium silicate-polymeric materials composite sheets. Mokuzaigakkaishi. 34 (10): 834–843 (in Japanese with English summary)

Segal, L., J.J. Crealy, A.E. Martin Jr. and C.M. Conrad 1959 Textile research J., 29 pp. 786.

Sobue, N., N. Hirai and I. Asano 1970 Studies on structure of wood by x-ray vibration of crystalline state in the stem of sugi. Mokuzaigakkaishi. 16 (6): 262–267 (in Japanese with English summary)

Wang, S. and C. Cho 1993 Equilibrium moisture contents of six wood species and their influences. Mokuzaigakkaishi. 39 (2): 126–137

Yamai, R. 1982 Mokuzaikoughou-handbook. Maruzen (Tokyo) pp. 100.