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Komoto, Masahiko
Fujii, Satoshi
Kishihara, Shiro
Yoshinaga, Kazuhiko

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STUDIES ON IMPROVEMENT OF QUALITY OF SAGO STARCH

Part I. Effect on the quality of some chemical and/or ultrasonic treatment

Masahiko KOMOTO*, Satoshi FUJII*, Shiro KISHIHARA* and Kazuhiko YOSHINAGA*

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Abstract

Commercial sago starch which is manufactured from pith of sago palm is cheap but inferior in the quality. Some properties of commercial sago starch were examined and for the improvement of its quality the starch was treated with chemicals and/or ultrasound.

1. The inferior quality of sago starch could be attributed mostly to the low whiteness.
2. The color of hydrolyzate of the sago starch was different from that of other kinds of starch.
3. Coloring matters in sago starch were constituted of both the soluble one and insoluble one in aqueous neutral solution. It seemed that the insoluble coloring matter largely brought on the low whiteness of sago starch.
4. Ultrasonication gave a remarkable effect on release of ash and flocculent matter from the sago starch. Treatment with DBS or Tween 60 was effective on release of the soluble coloring matter.
5. The flocculent matter was considerably released after sonication in the presence of Tween 60. It was found that the insoluble residue after hydrolysis of the sago starch was related to the flocculent matter.
6. The whiteness of the sago starch was improved largely after treatment with hydrogen peroxide or sodium hypochlorite.
7. X-Ray diffraction patterns did not show any change in the conformation of starch before and after treatment with chemicals and/or ultrasound.

Introduction

Eleven genera of the family Palmae and 3 genera of the family Cycadaceae are known to accumulate starch in their pithes[4]. Of these 2 species, *Metroxylon sagu* Rottb. and *M. rumphii* Mart. have high productivity of starch and are used as commercial souce of starch[6]. Present commercial sago starch comes principally from the pith of the 2 species of sago palm. Principal producers of sago starch are the East Indies, Malaya and Sumatra[11]. At present the production of sago starch is smaller than that of other kinds of starch. The production reached a peak in 1931 and declined thereafter[7].

Production of sago starch is principally native and export of the starch is not so large. Japan imported about 16,000 tons of sago starch from Sarawak in 1975[1]. Sago starch is manufactured mostly by hand labor and its cost is low but its quality is inferior. We suppose that the inferior quality is a principal cause of the low export. In recent years sago palm has been paid increasing attention and international sago syposia were held in 1976 (Sarawak) and 1979 (Kuala Lumpur)[6]. Few elemental reseaches on the production and utilization of sago starch, however, were published.

In the present paper some properties of commercial sago starch are compared with those of other kinds of starch. Furthermore the sago starch is treated with various

* Laboratory of Chemistry and Technology of Plant Products

chemicals including surfactants and/or with ultrasound to improve the quality.

Materials and methods

1. Sample starches

Sago starches (Sarawak 1979 and Sarawak 1981) produced in Sarawak in 1979 and 1981 were obtained from Matsutani Chemical Industry Co., Itami. Corn starch (C-starch) and potato starch (P-starch) were purchased from Nakarai Chemicals, Kyoto.

2. Chemicals

Polyoxyethylene sorbitan monostearate (Tween 60) was purchased from Nakarai Chemicals, Kyoto, sodium dioctylsulfosuccinate (SDS) and *n*-butylnaphthalenesulfonic acid sodium salt (BNS) from Tokyo Kasei Kogyo Co., Tokyo, sucrose fatty acid ester S-1170 (SFE) from Dai-Nippon Sugar MFG. Co., Tokyo, acetylated glycerin monostearate (Poem G-008) from Riken Vitamin Oil Co., Tokyo, and sodium dodecylbenzenesulfonate (DBS) and other chemicals from Wako Pure Chemical Industries, Osaka.

3. Chemical and ultrasonic treatment

Two grams of sample sago starch and 10ml of aqueous solution of various concentrations of chemicals were placed in a test tube (18mm ϕ). The sample was occasionally agitated by hand or continuously ultrasonicated. The sonication was carried out at 50°C for 2 hr with a sonifier (45 kHz, 50 W; Ultrasonic Cleaner B-12, Branson Cleaning Equipment Co., Connecticut). The agitation was carried out at room temperature for 2 days with hydrogen peroxide or sodium hypochlorite, and at 50°C for 2 hr with other chemicals.

Flocculent matter separated from the starch on top of the starch layer was observed with the naked eyes, and whiteness of the starch and color of the supernatant were measured. The amount of flocculent matter was shown by numbers of + mark, the whiteness (Hunter whiteness) was directly read with a color and color difference meter, Type ND-101 D, Nippon Denshoku Kogyo Co., Tokyo and the color of supernatant was expressed as the absorbance at 420nm.

The starch and supernatant were separated by the following manner. The supernatant was first separated from the starch by decantation. In decanting, the flocculent matter was transfered as quantitatively as possible to the supernatant part. The supernatant part was then centrifuged at 16,000rpm for 20min. The starch part was placed in a sintered glass filter, rinsed with water and air-dried.

4. Determination of protein

Total nitrogen content in sample starch was determined according to the Jaenicke's method[3] with 10-fold scale and multiplied by factor 6.25 to obtain protein content.

5. Ash

Ash was expressed as the residue after ignition at 550-600°C.

6. Total fat

Total fat was determined by an extraction method[10] using chloroform-methanol mixture.

7. Residue after hydrolysis

Ten grams of sample starch were placed in an Erlenmeyer flask of 300ml, and 10ml of 25% hydrochloric acid, 100ml of water and 1ml of amyl alcohol were added. The sample was refluxed for 30 min on a sand bath. The mixture was filtered through a sintered glass filter (3G-4) and rinsed with water. The residue was determined after drying to constant weight in an air oven at 105°C.

8. Color of solution after hydrolysis

Acid-hydrolysis

The color of the filtrate yielded in 7. was measured and expressed as the absorbance at 420 nm using a 1cm path length cuvette.

Enzymic hydrolysis

Suspension of 1g of sample starch and 4ml of α-amylase solution* were stirred for 1hr at 75°C and then boiled for 2min, followed by cooling to 50°C. In the case of C-starch this operation was again repeated after adding 4 ml of α-amylase solution, because the starch was not satisfactorily liquefied. To the mixture

* α-Amylase solution was prepared as follows. 80mg of industrial α-amylase was dissolved in 100 ml of 0.05 M phosphate buffer at pH 5.5 and filtered.

1ml of glucoamylase solution* was added and kept at 50°C for 2 days, followed by boiling for 2min. After adjusting the volume to 10 ml with water the mixture was centrifuged at 10,000rpm for 10min. The absorbance of the supernatant was measured at 420nm against a blank containing both the enzymes.

9. X-Ray diffraction

X-Ray diffraction pattern was recorded as the intensity of diffraction against the angle of diffraction with a X-ray diffractometer, Geigerflex RAD-IA, Rigaku Denki Co., Tokyo. Measurement was tried for moistened starch samples under conditions of X-ray : Cu/Ni 40kV 30mA, scan speed : 2°/min, full scale : 1 kcps, time constant : 2 sec, chart speed : 1 cm/min, slits : DS/SS 1° RS 0.3mm RSM 0.8mm and detector : SC.

10. Electrical conductivity

Conductivity was measured with a conductivity meter, CD-35MII, MS Instrument, Osaka.

Results and discussion

1. Properties of the sago starch

The X-ray diffraction pattern of sample sago starch is shown in Fig. 1. The obtained pattern (peak 1 is appreciable, 2a and 2b noticeable, 3a unnoticeable, 3b sharp, 4a and 4b strong, 5a appreciable, 5b faint, 6a strong, 6b and 6c noticeable, 7 appreciable and 8 noticeable) was assigned to C type.

Whiteness and impurities of the sago starch, together with those of C- and P-starch, are listed in Table 1. Appreciable difference in the whiteness between Sarawak 1979 and 1981 was observed and these whiteness were considerably lower than those of the other kinds of starch tested. The low whiteness is undesirable in the quality. The protein content of the sago starch was one half of that of C-starch and was slightly smaller than that of P-starch. The total fat content of the sago starch, together with that of P-starch, was very little. The low content of protein and total fat is desirable in the quality. The observed total fat content of C-starch was distinctly different from those described in literatures. Content of the total fat is probably varied according to the methods of analysis[10]. We suppose that difference in the analysing procedures perhaps contributed to the difference between the value in literature and observed one. The ash content of the sago starch was more than C-starch's and equal to P-starch's. The amounts of residue after hydrolysis of the sago starch were nearly the same as that of C-starch and were more than twice that of P-starch. The ash and the residue after hydrolysis of the sago starch were not especially much. The color intensities of acid hydrolyzate of the sago starch were about twice that of P-starch and were less than one half of that of C-starch. Enzymic hydrolyzate of Sarawak 1979 colored significantly, while that of C- and P-starch did not. The inferior quality of sago starch was thus clearly disclosed. We suppose that the stronger color resulted through acid-hydrolysis of C-starch was secondarily produced by reaction of the reducing sugar with amino acids which came from protein contained in the starch, as the color did not be formed through the enzymic hydrolysis at milder condition under which protein was not hydrolyzed. Since the enzymic hydrolyzate of C-and P-starch had no color, it seems that the color of enzymic hydrolyzate of Sarawak 1979 released from the original starch.

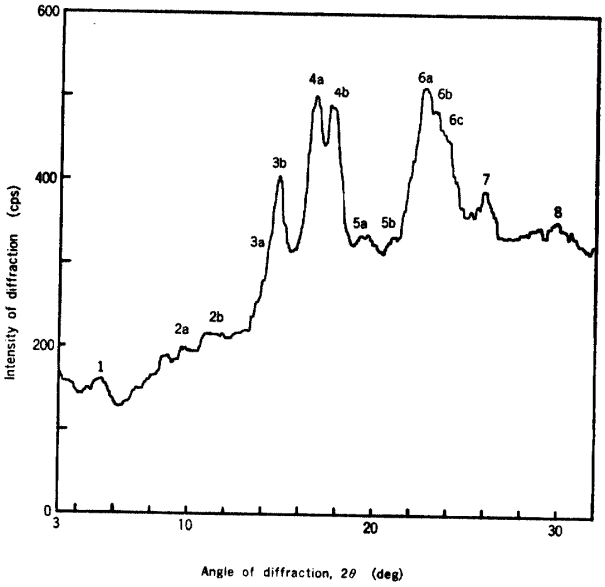

Fig. 1. X-Ray diffraction pattern of sago starch

* Glucoamylase solution was prepared as follows. 660mg of industrial glucoamylase was dissolved in 100ml of 0.05 M phosphate buffer at pH 5.5 and filtered.

Table 1. Whiteness and impurities of sample starches

	Sago starch observed 1979	Sago starch observed 1981	Sago starch in literature 2)	Sago starch in literature 9)	Corn starch observed	Corn starch in literature 9)	Corn starch in literature 5),8)	Potato starch observed	Potato starch in literature 9)	Potato starch in literature 8)
Whiteness (Hunter)	73.0	77.1	65–78	78.1	95.7	92–96		88.8	88–92	79–94
Protein (%)	0.15	0.15			0.3	0.25–0.35	0.25–0.35	0.25	0.04–0.08	0.06–0.12
Total fat (%)	0.04	0.09			0.26	0.05	0.05	0.02		
Ash (%)	0.23	0.34		0.11	0.05	0.06	0.06	0.23		
Residue after hydrolysis (%)	0.73	0.62			0.67			0.30		
Color of hydrolyzate (E_{420}) acid	0.09	0.07			0.22			0.04		
Color of hydrolyzate (E_{420}) enzyme	0.068				0			0		

Consequently, from the result given in Table 1 inferior quality of sago starch can mostly be attributed to the low whiteness as already Ito et al. pointed out[2].

When the acid-hydrolyzates of the sago starch were neutralized with sodium hydroxide solution, dark brownish red precipitate appeared but the color of the solution hardly changed, while in the acid-hydrolyzates of the other kinds of starch a precipitate hardly appeared and the color of solution raised greatly to about twice that before the neutralization. Moreover, the color of hydrolyzate of the sago starch was brownish pink and that of the other kinds of starch was yellowish brown. These results signify that the coloring matter of the hydrolyzate of sago starch is considerably different from hat of the other kinds of starch. The precipitate appeared during neutralizing the acid-hydrolyzate of the sago starch could be dissolved in 1 N sodium hydroxide. The residue after hydrolysis of the sago starch gave also dark brownish red and the coloring matter in the residue could also be dissolved in 1 N sodium hydroxide. The above results suggest that coloring matters in sago starch were constituted of at least two kinds of coloring matter. One of them is soluble in aqueous neutral solution and another, which is called insoluble coloring matter in this paper, is insoluble in neutral solution but soluble in alkaline solution. As the soluble coloring matter was not so much, it seems that the insoluble coloring matter is a main contaminant influencing on the whiteness of sago starch. We are trying to clarify the relation between the darkened color of sago starch, the precipitate appeared in neutralization of the acid-hydrolyzate and the coloring matter in the residue after hydrolysis.

2. Chemical and ultrasonic treatment

Sarawak 1979 treated with various chemicals and/or ultrasound are shown in Fig. 2 as the representatives. It was observed that a considerable amount of bulky flocculent matter appeared on top of the starch layer after treatment with 0.1% Tween 60 and sonication, and the supernatant was faintly browned. After sonication in the presence of 1% DBS or 1% Tween 60, the supernatant was noticeably colored. The color intensity of supernatant, the amount of flocculent matter and the whiteness of starch in all tests are listed in Table 2.

After treatment only with water the supernatant was scarcely colored, the flocculent matter hardly appeared and the whiteness remained unchanged. In the sample sonicated together with water, the flocculent matter noticeably appeared, but the color of supernatant was very faint and the whiteness of the treated starch hardly changed. Effect of treatment only with DBS and that of treatment with DBS and sonication were compared. The supernatant after sonication colored more strongly. The flocculent matter was appreciably separated after sonication but in the absence of sonication its amount was trace. Thus, it is apparent that the sonication gives somewhat good effect on the refining of the starch.

The whiteness of Sarawak 1979 was extremely

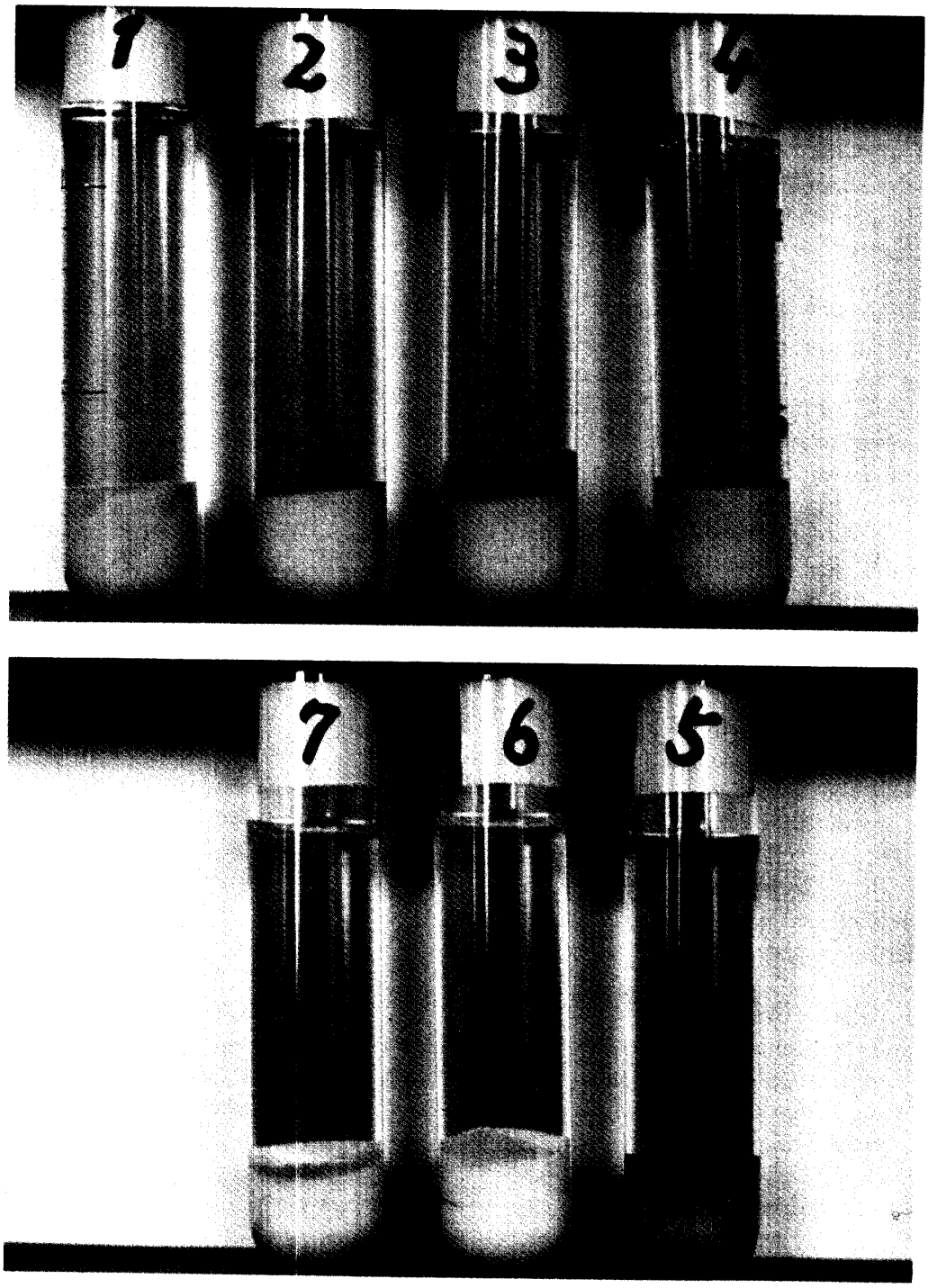

Fig. 2. Sago starch "Sarawak 1979" treated by sonication in the presence of some chemicals
1 : 0.1% DBS, 2 : 1.0% DBS, 3 : 0.1% Tween 60, 4 : 1.0% Tween 60, 5 : 0.1% Na_2SO_3, 6 : 0.63% H_2O_2, 7 : 0.12% NaClO

improved to 90.0 or 88.7 after treatment with 0.63% hydrogen peroxide or 1.2% sodium hypochlorite, respectively. In both cases the improvement of whiteness was probably caused by bleaching with the chemicals, because the higher the concentration of the chemicals, the less the color of the supernatant and the whiter the color of the starch. Other chemicals which brought out some good effect on the whiteness were DBS and Tween 60.

After the treatment with DBS or Tween 60 the coloring matter in the supernatant was perhaps released from the starch, since the supernatant was relatively much colored and the whiteness of starch became slightly higher. But the color intensity of supernatant was not always relative to the whiteness of starch. As all the color of the enzymic hydrolyzate may come from the soluble coloring matter in the starch, 70% or 65% of the soluble coloring matter would be released from the starch by the sonication in the presence of 1% DBS or 1% Tween 60, respectively. DBS or Tween 60 is thus effective for release of the soluble coloring matter from the sago starch.

Electrical conductivity of the supernatant was measured after the sonication- and water-treatment (Fig. 3). The conductivity increased almost linearly with the time of sonication. Relationship between conductivity and concentration of sodium chloride in aqueous solution was also shown in Fig. 3. The conductivity of supernatant after 4 hr sonication corresponds to that of 0.008% sodium chloride solution. The amount of sodium chloride in 10ml of the solution corresponds to 0.04% of the weight of sago starch tested and 17% of the weight of ash in the starch. This suggests that a considerable part of ash contained originally in the sago starch released by sonication.

The largest amount of flocculent matter was separated from the starch through the treatment with Tween 60 and sonication. After this treatment the flocculent matter part and starch part were separated from each other*, each part was hydrolyzed separately with 0.7 N hydrochloric acid and the insoluble residues

* Since flocculent matter could not completely be separated from the starch, the separated flocculent matter part included a considerable amount of the starch and the saparated starch part included a appreciable amount of the flocculent matter.

Table 2. Result of treatment with chemicals and/or ultrasound

Chemicals		Sonication*	Color of supernatant	Amount of flocculent matter	Whiteness
Water		×	0.013	+−	73.7
Water		○	0.018	+	74.0
McIlvaine's buffer	pH 3	○	0.037	+	73.7
	pH 4	○	0.047	+	73.8
	pH 5	○	0.080	++	73.9
	pH 6	○	0.097	++	74.6
DBS	0.1%	×	0.025	+−	—
	0.1%	○	0.037	+	75.4
	1.0%	×	0.032	+−	—
	1.0%	○	0.095	++	78.9
Tween 60	0.1%	○	0.055	+++	77.6
	1.0%	○	0.088	++	78.4
Poem	0.1%	○	—	++	76.8
BNS	0.1%	○	0.031	+	74.4
SDS	0.1%	○	0.030	+−	74.1
	0.25%	○	0.034	+	74.3
	0.5%	○	0.041	+−	75.0
	1.0%	○	0.037	+	76.2
SFE	0.1%	○	—	+	75.5
Na_2SO_3	0.1%	○	0.05	+	70.3
H_2O_2	0.06%	×	—	—	74.9
	0.16%	×	—	—	81.6
	0.32%	×	—	—	87.0
	0.63%	×	—	—	90.0
NaClO	0.012%	×	—	—	70.5
	0.06%	×	—	—	84.1
	0.12%	×	—	—	85.3
	1.2%	×	—	—	88.7

* Mark of ○ or × in the column of sonication means sonication or unsonication, respectively.

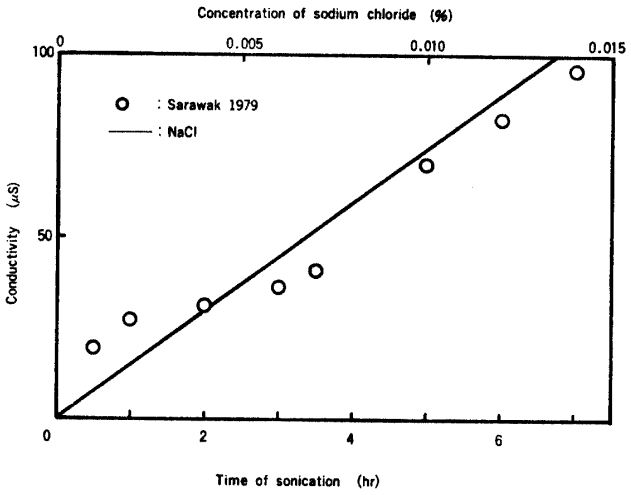

Fig. 3. Change in conductivity during sonication after treatment of sago starch "Sarawak 1979" with water and relationship beween conductivity and con centration of sodium chloride in aqueous solution

were determined. After hydrolysis of the flocculent matter part and the starch part, the insoluble residue per each original material was 8.2% and 0.53%, respectively. Since the sago starch untreated gave 0.73% of the residue, it seems that a considerable portion of the residue after hydrolysis consisted of the flocculent matter.

The sago starch treated with chemicals and/or ultrasound were subjected to X-ray diffraction. The patterns obtained were almost the same as that of the original starch. This shows that conformation of the starch did not changed by these treatments.

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サゴデンプンの品質改善に関する研究

第1報 薬品及び超音波処理の品質に及ぼす影響

河本 正彦・藤井 聰・岸原 士郎・吉永 和彦

要 約

サゴヤシから製造されたサゴデンプンは低価格であるが，品質が悪い。サゴデンプンの品質向上のための基礎的研究として，まず，サゴデンプンの2，3の性質を調べた。ついで，サゴデンプンを種々の薬品及び超音波で処理し，その品質の向上を試みた。

1．サゴデンプンの低品質は，主に，白度の低いことに帰因できた。
2．サゴデンプンの加水分解液の色は他の種類のデンプンのそれとは異なっていた。
3．サゴデンプン中の色素には中性水溶液に可溶性のものと不溶性のものがあり，サゴデンプンの低白度には不溶性色素が大きく影響しているものと思われた。
4．超音波処理は灰分及び絮状物をサゴデンプンから除去するのに非常に有効であった。DBS又はTween 60による処理は可溶性色素の除去に有効であった。
5．Tween 60—超音波処理によって，多量の絮状物がサゴデンプンから分離した。加水分解後の不溶性残渣はこの絮状物と関係があることがわかった。
6．サゴデンプンの白度は過酸化水素又は次亜塩素酸ナトリウム処理によって大幅に向上した。
7．薬品及び超音波処理前後で，デンプンの結晶構造に変化がないことを，X-線回折図によって確かめた。