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Study On The Variations In Characteristics Of Cassava Starch During The Chemical And Physical Modification Process Within Selected Locally Grown Varieties

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Abstract- Cassava is a potential source for starch. The study was conducted to improve the functional properties of native starch through hydrothermal and cross-linking modifications. The starch was extracted from SWARNA, KIRIKAWADI, MU51, SHANI, JS1, 565 and extracted starch was dried at 55°C. The physical characteristics were analyzed of native and modified starches. The highest starch yield was observed in MU51 variety (15.25%) and had more export potential. The hydrothermal modification was carried out for MU51 and SHANI varieties. The modified starch had a significant reduction in solubility (0.22±0.65%, 0.83±0.08%) ($p<0.05$). The swelling power (253.83±0.014%, 214.08±0.26%) and water absorption capacity (113.77±0.67%, 100.09±0.75%) of the modified starch was significantly higher than the native starch. the dual-modified starches had the highest Gelatinization Temperature (68.00±0.00°C, 70.00±0.00°C) than native starches ($P<0.05$. The hydrothermal modified starches had a significant reduction in viscosity. The crosslinking modification was carried out for the MU51 with different concentrations of cross-linking agents (STMP/STPP) (0%, 6%, and 12%). The solubility of cross-linked cassava starch with 12% concentration (4.37±0.28%) was higher than native starch ($p<0.05$). The swelling power of cross-linked cassava starch with 12% concentration (222.94±0.54%) was higher than other samples ($p<0.05$). the native starch had the lowest Gelatinization temperature value (67.00±0.00°C) than cross-linked cassava starch ($p<0.05$). The cross-linked starch with 12% concentration had the highest Foaming capacity (1.96%) ($p<0.05$) and a decrease in viscosity when compared to native starch. The result suggested that, the hydrothermal and cross-linked modified starch are suitable for the harsh conditions in food processing technology than native starch.

Keywords: Cassava starch, Cross-linking modification, hydrothermal modification

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1. Introduction

Cassava (*Manihot esculenta Crantz*) belongs to the Euphorbiaceae family and has tuberous roots. Cassava, originally from South America, is now widely grown in Asia's tropical and subtropical regions (Zhu, 2015). Cassava root yields vary with variety (MU51, SAHANI, KIRIKAWADI, 565, JS1, SURANIMALA), plant growth conditions such as soil, climate, and rainfall, and agronomic practices including fertilizer application, weed control, irrigation, and multiple cropping (Breuninger et al., 2009*)*. Sri Lanka has several kind of cassava verities such as MU51, SAHANI, KIRIKAWADI, 565, JS1, SURANIMALA and more potential for cassava production. Cassava is the best source for starch production and has the potential to produce modified starch. According to the Department of Statistics, 307,996mt of cassava will be produced in 2022, with 1,401,538kg of cassava starch imported in 2023 in Sri Lanka.

Starch occurs in the form of tiny white granules in various sites of plants, for example in cereal grains (maize, rice, wheat, barley, oat, sorghum), in roots (sweet potatoes, cassava, arrowroots, yam), in tubers (potatoes), in stems (sago palm) and in legume seeds (peas, beans). Starch contains a linear molecule termed amylose and a highly branched polymer termed amylopectin. Potato (21%) and tapioca starch (17%) have a much lower amylose content as compared to 28% for maize and wheat starch (Swinkels, 1985).

Native starches have limitations in food processing, including loss of viscosity and thickening power during cooking and storage, especially at low pH, as well as retrogradation and syneresis. Modified starches are intended to address one or more of the common limitations of native starch. Physical, enzyme, biological, and chemical modifications are all used to alter starch properties. Chemical modification is the most widely used process in the industry (Gunorubon & Kekpugile, 2012*)*. In this method, native starch is treated with various chemicals under specific operational parameters. The cross-linked method is one of the best chemical modification methods of starch. It increases the amylopectin content of native starch and makes the starch more stable (Gunorubon & Kekpugile, 2012). Many studies were investigated about starch modification, there is lack of information on modification of starch, from locally grown cassava varieties and physical properties of native and modified starches. The Objective of this study is to determine the starch characteristics of six locally grown cassava varieties and to develop a modified cassava starch that will exhibit improved physical properties. In addition, to identify the starch characteristics differences between native cassava starch and its modified version.

2. Materials and Methods

2.1. Materials

The cassava samples from selected varieties (SHANI, SWARNA, JS1, 565, KIRIKAWADI,) were purchased from the farmers certified by the Department of Agriculture, Sri Lanka. Required Chemical compounds and other required types of machinery and equipment were obtained from food section laboratories in Industrial Technology Institute (ITI).

2.2. Extraction of starch from different varieties of cassava tubers.

The cassava samples were collected and cleaned to remove the outer foreign materials and the peel was removed. Cassava tubers were washed 3 times with potable water. The cassava tubers were cut into small pieces and put into the water bath until crushed to reduce the browning. The cassava pieces were ground while adding water using an electric Blender (Japan, family mate). Blended samples were filtered using a nylon cloth separately into the beaker (5000ml). In addition, remain sample (in nylon cloth) was washed with water in a water bath and kept it 24hr to settle the starches. The starch was separated from the water and repeated the washing and settling process several times to remove impurities. Settled fresh starch samples were put into the dehydrator trays for air drying. The fresh starch samples were air dried at 55 ℃ for 5hr by a heat pump dryer (Nesco professional, America) and dried samples were separately ground using electrical blender (family mate, Japan) The ground sample were sieved using 150µm sieve and kept in sealed poly propylene bags at room temperature until further testing.

2.3. Preparation of cassava cross-linked modified starch

The cassava cross-linked modified starch was prepared using the method described by Li et al., 2022, with minor modifications. 40 g of native starch (MU51) was dispersed in 60 mL of water, and 4 g of Na2SO4 was added and thoroughly mixed. The solution's pH was adjusted to 11.5 using NaOH (1 mol/L).Three solutions were prepared separately by mixing 0%, 6%, and 12% (w/w) of cross-linking reagents (STMP/STPP) independently, according to a 99:1 ratio respectively. The solutions were placed in a water bath and stirred at 50°C for 4 hours. The pH was adjusted to 6.5 using HCl (1mol/L) and the starch slurry was washed three times with distilled water until neutralized (7.13 pH). The pH was determined with pH meter ((Eu Tech pH/mv/ºC, Singapore). Then, the prepared solutions were centrifuged at 3000 RPM for 20 minutes using refrigerated centrifuge. The centrifuged samples were air dried at 45◦C for 24 hours using dehydrator (Nesco professional, American) and dried samples were separately ground using an electric Blender (family mate, Japan). The ground samples were sieved using a 150 µm sieve and kept in sealed polypropylene bags until further testing.

2.4. Preparation of hydrothermally modified cassava starch

The native cassava starch samples from the SHANI and MU51 varieties were subjected to one-step annealing according to a method of Ariyantoro et al., 2022 With slight modification. First, 60 g of the native starch was weighed and mixed with 180 ml of distilled water (ratio 1:3) in a sealed conical flask. The samples were incubated at 55°C for 24 hours using a shaking water bath (Memmert, Germen). After that, incubated samples were decanted and air-dried at 40°C for 24 hours using dehydrator (Nesco professional, American). The dried samples were separately ground using an electric Blender (Family mate, Japan). The ground samples were sieved using a 150 µm sieve and kept in sealed polythene bags. The sieved sample was considered as single-annealed cassava starch.

Then the hydrothermal modification process was continued using heat moisture treatment (HMT). The HMT treatment was carried out according to the method described by Ariyantoro et al., 2022 with slight modification. The 40 g of annealed starch from the SHANI and MU51 varieties were accurately measured into Petri dishes, and the moisture level was adjusted to 30% by adding the appropriate quantity of distilled water. The Petri dishes were sealed and subjected to incubation at 4°C for 24 hours in a refrigerator (Samsung RS72R5001M9/G). The incubated starch samples were oven dried at 110°C in oven (Memmert, Germen) for 16 hours and this heat treated samples were air dried at 40°C for 24 hours. Finally, dried samples were separately ground. The ground samples were sieved using a 150 µm sieve and kept in sealed polypropylene bags.

3. Sample Analysis

3.1. Swelling capacity (SC) and solubility (S)

Determined the solubility and swelling power of native and modified cassava starch according to the method of Shad et al., 2013 with slight modification. 1g of starch was weighed into a falcon tube and dispersed with 50 mL of distilled water. The solution was heated at 60°C for 30 minutes in a thermally controlled shaking water bath (Memmert, Germen). The heated starch sample was allowed to cool to room temperature and it was centrifuged at 1500rpm for

15 minutes. The residue obtained from centrifugation, along with the water it retained, was weighed.

$$\boxed{\text{SC}\ (\%) = \frac{WrW}{WS} \times 100} \quad (01)$$

WRW= weight of the residue along with the water
WS = weight of the sample

The supernatant was collected into the pre-weighted metal moisture dishes. The supernatant was evaporated at 110°C in a thermally controlled water bath (Memmert, Germen). The evaporated dishes were oven-dried at 105°C for 2 hours in a drying oven (Memmert, Germen). Afterward, the residue was weighed and the present solubility of starch in water was calculated as:

$$\boxed{S(\%) = \frac{Wr}{WS} \times 100} \quad (02)$$

Ws = weight of the sample
Wr = weight of the residue, after the evaporation

3.2. Determination of Gelatinization Temperature

The gelatinization temperature was determined according to a method of Gunorubon & Kekpugile, 2012 with slight modification. The 1.5g of starch sample was accurately weighed and 10 ml of distilled water was added. The starch was dissolved in a borosilicate beaker. The solution of starch was stirred and the thermometer was placed in a beaker. The beaker was placed in a thermally controlled shaking water bath. The solution was stirred continuously until it became milky and thick. That was the gel point, and the temperature there was recorded as the sample's gelatinization temperature.

3.3. Determination of Viscosity

The viscosity profile was determined using viscometer. 20g of starch sample was weighed into a borosilicate beaker (250 ml) and dissolved with 200 ml of distilled water. The readings were obtained under the different temperature (room temperature, 50°C, and 50°C cool into room temperature, 70°C, and 70°C cool into room temperature). Afterwards, the spindle numbers 02 and 06 (according to the condition of solution) were set at speeds the rate of 60 and 100rpm and the reading was taken under both speeds.

3.4. Water absorption capacity (WAC)

The WAC was obtained according to method from Shad et al., 2013. The 1g starch sample was weighed into a pre-weighed falcon tube and mixed with 10 mL of distilled water. The solution was vortexed for 5 minutes using vortex mixer (Zx^3, Italy). The vortexed sample was centrifuged at 3000 rpm for 15 minutes using a refrigerate centrifuge. The supernatant was removed, and the sediments were weighed. Finally, the WAC is obtained as:

$$\boxed{WAC(\%) = \left[\frac{\text{W2} - \text{W1}}{\text{W0}}\right] \times 100} \quad (03)$$

W0 = Weight of the sample

W1 = Weight of the falcon tube
W2 = Falcon tube + sediments

3.5. Foaming capacity (FC)

Both native and modified starches were tested for foaming capacity (FC). The FC was determined using the method of Omojola et al., 2010 with slight modification. The 1g of starch sample was weighed into a 50 ml of falcon tube and dispersed with 50 ml of distilled water. The volume of solution was recorded by 100 ml of measuring cylinder. Afterward, the solution was homogenized for 5 minutes using vortex mixer (Zx^3,Italy). The homogenate was poured into a 100 mL measuring cylinder, and the volume was determined after 30 seconds. The FC was expressed as a percentage increase in volume.

$$\boxed{FC\ (\%) = \left[\frac{\mathrm{W2} - \mathrm{W1}}{\mathrm{W0}}\right] \times 100} \qquad (04)$$

W0 = sample weight
W1 = the volume before homogenized
W2 = the volume after homogenized

3.6. Emulsion capacity (EC)

The EC was determined according to method of Omojola et al., 2010 with slight modification. First, 1g of starch sample was weighed into a 25 ml of falcon tube and dispersed with 12.5 ml of distilled water. The solution was homogenized for 30 s using vortex mixer (Zx^3, Italy). The 12.5 ml of vegetable oil (peanut oil) was added gradually and homogenized for 30 s again. After homogenization, the solution was centrifuged at 3000 rpm for 15 minutes in a refrigerate centrifuge. The volume of the oil layer separated from the sample was obtained directly from the tube.

3.7. Statistical analysis

Differences between physical characteristics of native starches and modified starches were compared using analysis of variance (ANOVA) followed by Tukey's HSD post-Hoc Test. SPSS version 22 was used as the statistical software. Results were considered significant if the p-value is < 0.05.

4. Results and Discussion

4.1. Starch Extraction results from different varieties of cassava tubers.

Starch was extracted from six varieties of cassava tubers, and the properties of fresh and dry starch yield were outlined in Table 1.

Table 1

Starch yield from different varieties of cassava.

Cassava variety	Fresh starch weight (%)	Dry starch weight (%)
C1(SHANI)	13.6	11.3
C2(SWARNA)	14.7	9.28
C3 (JS1)	12.4	6.72
C4 (565)	14.2	11.6
C5 (MU51)	26.6	15.25
C6 (KIRIKAWADI)	16.6	9.44

The fresh starch was obtained from different varieties of cassava roots. Among those varieties the C5 (MU51) sample was observed high yield (26.6 %) than other varieties and lowest yield was observed in C3 (JS1) variety (12.4%). The yield of dry starch was obtained after the dehydration process. Among those samples, the C5 (MU51) sample was observed high yield (15.25%) and lowest yield observed in C3 (JS1) variety (6.72%).

4.2. The physical properties between native and hydrothermal modified starch

4.2.1. Solubility (S)"

The results showed that the native starch of C1 (SHANI) and C4 (MU51) had a solubility value of 0.22±0.06, 0.83±0.08 respectively. While the dual modified starch of CP1 and CP4 (SHANI and MU51) had solubility of about 0.11±0.02 and 0.55±0.28 respectively. The result showed that the native starches (C1, and C4) had the highest solubility (0.22±0.06, 0.83±0.08 respectively) than dual modified starches (CP1, and CP4). The solubility of native and hydrothermal modified cassava starches was presented in figure 1.

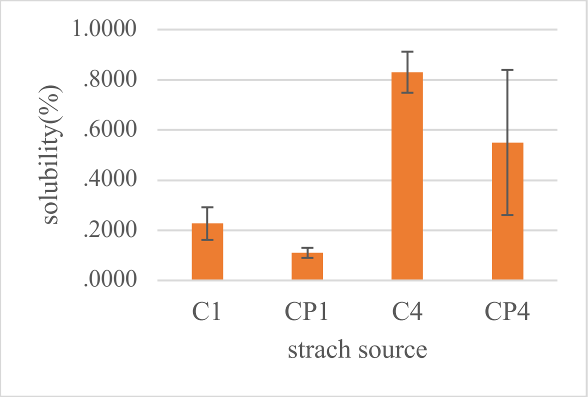

Figure 1. *The mean of solubility between native and hydrothermal modified starches*

4.2.2. Swelling capacity (SC)"

According to the statistical analysis, the native starches (C1, and C4) had the lowest swelling power (229.54±0.11, and 197.86±1.91 respectively) than dual modified starches (CP1, and CP4) (253.83±0.01, and 214.08±0.26). The SC of native and hydrothermal modified cassava starches were presented in figure 2.

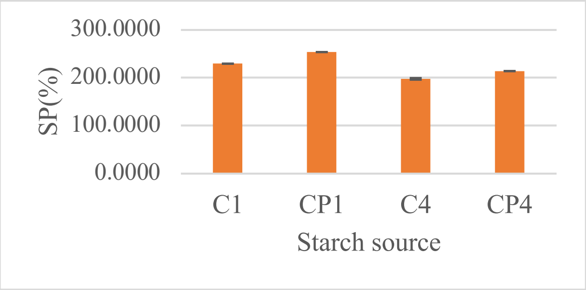

Figure 2. *The mean of swelling power between native and hydrothermal modified starches*

4.2.3. Water absorption capacity (WAC)"

The high water ratio and temperature of the annealing process increased the water absorption capacity. Furthermore, the HMT process expanded the amorphous region and make the water easily penetrate by breaking the hydrogen bonds. Thus, increased the water absorption capacity of starch (Ariyantoro et al., 2022). The statistical results, indicate that the WAC of hydrothermal-modified starches of CP1, and CP4 was higher than the natural starch.

4.2.4. Gelatinization Temperature (GT)

The starch forms a transparent gel, that point is known as the gelatinization temperature. It is the process of breaking down the intermolecular bonds of starch granules by heat and water. That process allows to engage more water (Swinkels, 1985). The result showed that the native starches (C1, and C4) had the lowest GT (65.00±0.00, and 67.00±0.00 respectively) than dual modified starches (CP1, and CP4) (68.00±0.00, and 70.00±0.00). The GT of native and hydrothermal modified cassava starch were presented in figure 3.

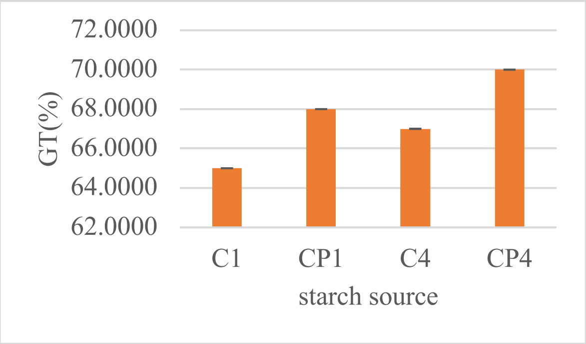

Figure 3. *The mean of GT between native and hydrothermal modified starches*

4.2.5. Foaming capacity (FC)"

The flexible protein molecule affects the foaming characteristics and it influence on surface tension of water. The foaming properties are important to maintain the structure of food products (Shad et al., 2013). According to the results, the foaming capacity was not observed in both native and hydrothermal modified starches.

4.2.6. Viscosity"

Table 2
The viscosity values between native and hydrothermal modified starch

Sample	temperature°C	Viscosity at 60rpm	Viscosity at 100rpm
C1	50	8.63±0.05^{a}	10.03±0.05^{a}
CP1	50	7.40±0.00^{b}	10.26±0.05^{a}

C4	50	7.36 ± 0.05^{b}	9.73 ± 0.05^{b}
CP4	50	6.72 ± 0.20^{c}	9.33 ± 0.15^{c}

Table 2 presents the viscosity values of native and hydrothermal-modified starches. The C1 and C4 sample (native) were observed highest amount of viscosity than CP1 and CP4 sample (modified). The viscosity was reduced by hydrothermal modification than native starch.

4.3. The physical properties between native and cross-linked modified starch

Table 3

The results of physical characteristics between native and cross-link modified starches

Sample name	**Solubility (%)**	**Swelling power (%)**	**GT (%)**	**WAC (%)**	**FC (%)**	**EC**
C4	0.83 ± 0.08^{b}	197.86 ± 1.91^{b}	67.00±0.00	66.10 ± 0.83^{d}	0.00 ± 0.00^{c}	Nil
0%	0.62 ± 0.02^{b}	194.03 ± 8.23^{b}	70.00±0.00	88.57 ± 0.33^{a}	0.00 ± 0.00^{c}	Nil
6%	0.49 ± 0.02^{b}	189.39 ± 5.29^{b}	Nil	82.88 ± 0.30^{b}	0.98 ± 0.00^{b}	Nil
12%	4.37 ± 0.28^{a}	222.94 ± 0.54^{a}	Nil	78.33 ± 0.28^{c}	1.96 ± 0.84^{a}	Nil

4.3.1. Solubility

According to the statistical results, it indicates that the solubility of cross-linked cassava starch with 0%, and 6% concentration was lower than native starch but the cassava cross-linked starch with 12% concentration was higher than native and other samples (0%, and 6%). The solubility level of native and cross-linked cassava starches were presented in figure 4.

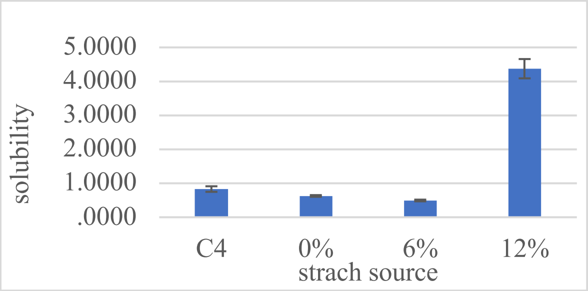

Figure 4. *The mean of solubility between native and cross-linked modified starches*

4.3.2. Swelling capacity (SC)

According to the statistical results, it indicates that the swelling power of cross-linked cassava starch with 0%, and 6% concentration was lower than native starch but the cross-linked starch with 12% concentration was higher than native and other samples (0%, and 6%). The swelling power of native and cross-linked cassava starch were presented in figure 5.

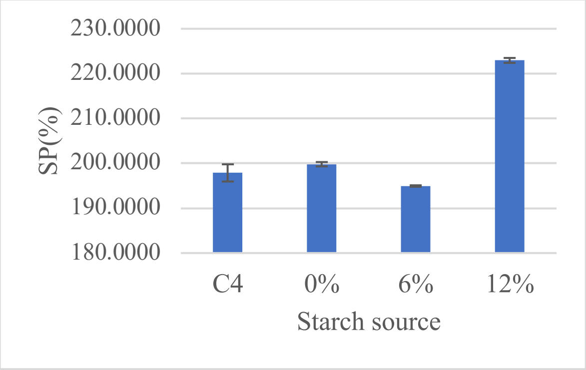

Figure 5. *The mean of swelling capacity between native and cross-linked modified starches*

4.3.3. ***Gelatinization Temperature (GT)***

The C4 sample had lowest GT value than 0%, 6%, and 12% samples. According to the method of determination of GT, the sample was heated only up to 100 °C. But, the gelatinization point of 6%, and 12% samples were heated higher than 100°C. Because of that, the gelatinization temperature was not detectable of 6%, and 12% samples.

4.3.4. ***Water absorption capacity (WAC)***

The water absorption is the amount of water needed for starch to swell. It is expressed the percentage of water that absorbed by starch. According to the statistical results, it indicates that the WAC of cross-linked cassava starch with 0%, 6%, and 12% concentration was higher than the native starch.

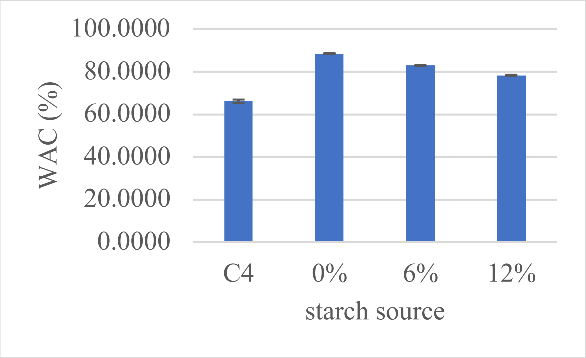

Figure 6. *The mean of WAC between native and cross-linked modified starches*

4.3.5. ***Foaming capacity (FC)***

The results showed that the FC of C4 (native MU51), and 0% were not observed. While the cross-linked modified cassava starch of 6%, 12% had FC of about 0.98±0.00 and 1.96±0.84% respectively. According to the statistical results, it indicates that the FC of cross-linked cassava starch with 6%, and 12% concentration was higher than the native starch, and 0% sample.

*4.3.6. **Viscosity***

Table 4

The viscosity values between native and cross-linked modified starch

Sample	temperature°C	Viscosity at 60rpm	Viscosity at 100rpm
C1	50	7.36±0.05c	9.73±0.05c
CP1	50	7.30±0.10c	10.30±0.10b
C4	50	7.96±0.05a	10.23±0.05b
CP4	50	7.70±0.00b	12.80±0.00a

Table 4 presents the viscosity values of native and cross-linked modified starches. The viscosity was significantly different within the treatments. The C4 sample was observed highest viscosity than modified starches (0%, 6%, and 12%). The 12% sample was observed lowest viscosity than other samples. The results showed that, the cross-linking modification was reduced viscosity. Therefore, the cross-linked starches can be used for harsh condition in food processing operation.

5. Conclusion

The results showed that the hydrothermal modified and cross-linked modified starch suitable for the harsh conditions in food processing technology than native starch. The MU51 variety was showed high fresh and dry starch yield than other varieties. The MU51 is suitable to cross-linked and hydrothermal modification. The high solubility was observed in cross-linked starch (MU51) than hydrothermal modified starch. The highest swelling capacity was observed in cross-linked starch than hydrothermal modified starch. The water absorption capacity was increased by both cross-linked and hydrothermal modification methods. The highest gelatinization temperature was observed in cross-linked modified starch. The cross linked starch is suitable for modern food processing operation than hydrothermal modification.

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