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Gelatinization Properties and Bread Quality of Flours Substituted with Hydroxypropylated, Acetylated and Phosphorylated Cross-linked Tapioca Starches

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Abstract: Three kinds of chemically modified tapioca starches, *i.e*., hydroxypropylated tapioca starch (HTS), acetylated tapioca starch (ATS) and phosphorylated cross-linked tapioca starch (PTS), were substituted for 20% (w/w) of wheat flour, and gelatinization and breadmaking properties of the substituted flour were compared with those characteristics of the flours with native tapioca starch (NTS) or without any tapioca starches (control). Gelatinization temperatures (T_{o1}, T_{p1} and T_{f1}) and endothermic enthalpy change (ΔH_1) of the dough with 20% of HTS were quite similar to those of the control on DSC curves. NTS, HTS and ATS swelled and collapsed easily during heating as compared with wheat starch, while PTS hardly swelled and was difficult to disperse as compared with NTS, HTS, ATS and wheat starch. The substituted flours except HTS significantly decreased water absorption measured by a Farinograph as compared with the control. With regards to the mixing times obtained from a Farinograph, doughs made from the substituted flours were shortened, but the mixing tolerance was the same as compared with the control. Specific volume of loaves baked from the substituted flours decreased distinctly, but that of HTS was the largest among the substituted samples. The bread crumbs baked with HTS were softer than those with NTS or the control during storage, whereas bread crumbs with ATS or PTS were harder. Among the three kinds of chemically modified tapioca starches, HTS was the most suitable substitution for wheat flour in breadmaking.

Key words: hydroxypropylated tapioca starch, acetylated tapioca starch, phosphorylated cross-linked tapioca starch, bread, dough

Recently chemically modified starches such as esterified, etherified and cross-linked starches have been widely employed for various prepared foods such as snack foods, bread, and cakes to improve their quality.[1–4)]

Esterified and etherified starches possess many similar properties, such as low gelatinization temperature, paste clarity, lower gel forming ability, higher freeze-thaw stability and higher water-holding properties.[5–9)] When a reagent that has two or more reactive functional groups is reacted with starch chains, intra- or intermolecular cross-linkages are commonly formed. Cross-linking alters starch properties by increasing the pasting temperature, suppression of swelling of starch granules during cooking to obtain desirable food texture, increasing and/or stabilizing viscosity in severe cooking or in conditions of high shearing and acidic pH, improving freeze-thaw stability and water-holding properties, and reducing clarity of starch pastes.[4,7,10,11)]

Bread is a bakery product that contains higher amount of starch. Some workers have studied bread quality by substituting chemically modified starch for wheat starch[12,13)] or wheat flour.[14,15)] Inagaki and Seib[12)] and Toufeili *et al*.[13)] studied bread staling with cross-linked waxy barley starch. They reported that bread with cross-linked waxy barley starch staled easily and showed a higher enthalpy change of melting of crystalline region of starch. However, Hung and Morita[14)] suggested that dough substituted with an optimum amount of cross-linked corn starches for wheat flour had higher resistance to extension and higher extensibility, and the bread showed bigger loaf volume. Takasaki and Mineki[15)] reported that bread containing 30% (w/w) of cross-linked hydroxypropylated potato starch showed softer crumbs than that containing native potato starch. However, there are few research papers on the quality of bread with chemically modified tapioca starches.

In the present study, three kinds of chemically modified, *i.e*., esterified, etherified and cross-linked tapioca starches were substituted for wheat flour, and the gelatinization properties and bread quality of the substituted flour were evaluated.

MATERIALS AND METHODS

Flour and starches. The wheat flour was commercial flour 'Hermes' (Okumoto Flour Milling Co., Ltd., Osaka, Japan). The protein and ash contents were 11.8 and 0.38 % (w/w), respectively, on a 13.5% (w/w) moisture basis. Three kinds of modified starches (Nippon Starch Chemi-

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cal Co., Ltd., Osaka, Japan) were used as follows: **I.** Hydroxypropylated tapioca starch (HTS) was "G-800" with substitution percent of around 3.1–3.9 {degree of substitution (DS) 0.09–0.11}. **II.** Acetylated tapioca starch (ATS) was "Z-300F" with substitution percent of 0.8–1.0 (DS 0.03–0.04). **III.** Phosphorylated cross-linked tapioca starch (PTS) was "PB-7000" with substituted phosphorus percent of 0.015–0.02. Native tapioca starch (NTS, Nippon Starch Chemical Co., Ltd., Osaka, Japan) was used for the reference. Commercial powdered gluten, "Fresh dry gluten", was obtained from Asama Chemical Co., Ltd. (Tokyo, Japan).

Physicochemical properties of dough.

Mixing properties of flour. Twenty percent of tapioca starches containing 8% (w/w) of vital gluten were substituted for wheat flour. Water absorption, arrival, development times and breakdown of doughs prepared from the substituted flours were determined by the Farinograph (Brabender Instruments, Inc., Germany) equipped with a 50 g stainless steel bowl according to the AACC Approved Method 54-21.[16] Mixing was performed in duplicate.

Dough preparation. The substituted flour and distilled water was mixed with a mixer KN-200 (Taisho Denki Co., Ltd., Shiga, Japan) for 15 min at the speed of 380 rpm. The amount of added water was determined from Farinograph water absorption. Temperature of the water added was controlled so that the final temperature of the mixed dough became 30 ± 1°C.

Gelatinization properties of the substituted flour and dough. Pasting properties of the substituted flour were tested using an Amylograph PT 100 (Brabender Instruments, Inc.) according to the AACC Approved Method 22-10 and 61-01.[17,18] In brief, 65 g of the substituted flours was homogeneously dispersed in 450 mL of distilled water. The suspension was heated from 35°C to 95°C at a rate of 1.5°C/ min, and held for 15 min at 95°C, cooled to 35°C at a rate of 1.5°C/ min, and held for 15 min at this temperature with continuous stirring at 75 rpm. Measurement was performed in duplicate.

Thermal characteristics of the dough made from the substituted flour were determined with DSC apparatus (Model DSC-60, Shimadzu Co., Ltd., Kyoto, Japan), controlled by TA-60 WS software and connected to a thermal analyzer.[19] The dough (11–15 mg) was weighed into an aluminum pan and hermetically sealed. Measurements were performed from room temperature to 120°C at a rate of 5°C/min under nitrogen purge gas. The results are presented as an average and standard deviation of three measurements.

Breadmaking. Bread was prepared from the substituted flour according to the previous method.[20] The substituted flour (300.0 g), sugar (15.0 g), salt (4.5 g), dry yeast (3.6 g) and an optimum amount of water (determined by the Farinograph) was mixed in a Ladies mixer KN-200 (Taisho Denki Co., Ltd., Osaka, Japan) for 15 min at the speed of 380 rpm. The kneaded dough was subjected to fermentation at 30°C and 85% relative humidity (RH) for 90 min. Knock-back was performed after 60 min in a fermentation stage. The fermented dough was divided into 130-g pieces and rounded to rest for 20 min at room temperature. The rounded doughs were sheeted and rolled by a SM-230 molder (Baker's Production Co., Ltd., Osaka, Japan). After that, the dough was placed into aluminum pans (upper size, W 105 mm×D 60 mm×H 69 mm; lower size, W 91 mm×D 55 mm×H 62mm) and proofed at 38°C and 90% RH for 50 min, followed by baking at 200°C for 15 min.

The baked loaves were weighed and the volume was measured by a rapeseed displacement method after cooling for 30 min at room temperature.

Firmness of the bread crumbs was measured by a rheometer (RT-2002 D·D, Rheotec Co., Ltd., Tokyo, Japan) as described previously.[21] The loaf was sliced into 30 mm of thickness, and the slice was cut into W 50 mm ×D 50 mm×H 30 mm. The sample was compressed 7 mm of deformation with a plunger (ϕ20 mm×H 2 mm) at 5 mm/s of compression speed. Data were processed using the Rheosoft TR-06 computer program (Rheotec Co., Ltd.). The results are presented as an average of six slices of crumb. The crumbs were wrapped up into polyethylene bag and stored at 22°C.

Statistical analysis. Analysis of variance (ANOVA) was evaluated by Duncan's multiple-range test ($p < 0.05$) using SPSS (Version 11.0, SPSS Inc., Chicago, IL, USA) to compare treatment means.

RESULTS AND DISCUSSION

Mixing properties of dough prepared from the substituted flours.

Figure 1[22] shows a typical Farinogram. This chart informs us of water absorption of flours and physical stability and other mechanical characteristics of doughs during mixing. A, E, and F in Fig. 1 are defined as the development time, arrival time and time to breakdown, respectively. In general, the strong type of wheat flour shows longer arrival and development times and time to breakdown. Water absorption, to be brief, is an appropriate percentage of water added to constant weight of flour. Water added is controlled so that a center of the mixing curve at maximum consistency becomes 500 B.U.

Mixing properties of the flours substituted with native or various modified tapioca starches for 20% (w/w) of wheat flour are summarized in Table 1. Substitution of modified tapioca starches except HTS decreased the water absorption. Water absorption of HTS dough was the same as compared with the control. Ono *et al*.[23] reported that

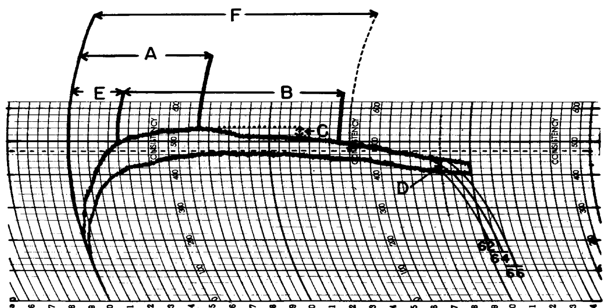

Fig. 1. Readings commonly used in interpreting a Farinogram.[22]

A, development time; B, stability; C, mechanical tolerance index; D, valorimeter value; E, arrival time; E+B, departure time; F, time to breakdown.

Table 1. Mixing properties of the flour substituted with native or various modified tapioca starches for 20% of wheat flour measured with a Farinograph.

Tapioca starch	Water absorption (%)	Arrival time (min)	Development time (min)	Time to breakdown (min)
Control	65.0	3.3	14.0	35.2
20% NTS	61.8	1.2	2.0	31.5
20% HTS	65.3	1.9	7.8	32.5
20% ATS	61.0	1.4	2.5	33.0
20% PTS	63.5	1.1	1.8	34.8

NTS, native tapioca starch; HTS, hydroxypropylated tapioca starch; PTS, phosphorylated cross-linked tapioca starch; ATS, acetylated tapioca starch.

Table 2. Pasting properties of the flour substituted with native or various modified tapioca starches for 20% of wheat flour measured with an Amylograph.

Tapioca starch	Pasting temperature (°C)	Peak viscosity temperature (°C)	Peak viscocity (B.U.)	Breakdown viscocity (B.U.)	Final viscocity (B.U.)	Setback (B.U.)	Total Setback (B.U.)
Control	61.3	91.6	594	181	1394	801	982
20% NTS	63.5	89.8	640	244	1258	618	862
20% HTS	58.3	90.3	630	232	888	258	489
20% ATS	62.2	90.4	655	236	1064	409	645
20% PTS	59.8	91.5	393	100	891	498	598

Abbreviations are the same as in Table 1. B.U., Brabender unit.

substitution of hydroxypropylated wheat starch for wheat flour increased water absorption evaluated with a Farinograph as compared with wheat flour. Takasaki and Mineki[15] reported that substitution of hydroxypropylated cross-linked potato starch for 10–30% (w/w) of wheat flour decreased water absorption as compared with wheat flour, whereas the same substitution of native potato starch increased the value. Higher water absorption of the flour with hydroxypropylated tapioca starch than that with native tapioca starch was consistent with the results of Takasaki *et al.*[15] and Ono *et al.*[23]

Arrival and development times of substituted flours were remarkably reduced as compared with the control. Shorter mixing time of substitution of native tapioca starch for wheat flour was consistent with the results obtained by Keya and Hadziyev[24] and Defloor *et al.*[25] In this study, substitution of HTS, ATS and PTS for 20% (w/w) of wheat flour also reduced the mixing time.

Times to breakdown of all substituted flours were almost the same as the control. This means substitution of native or chemically modified tapioca starches for 20% (w/w) of wheat flour has no deterioration to mixing tolerance.

Pasting properties of substituted flours.

Pasting properties of the substituted flours during the heating and cooling process are shown in Table 2. Peak viscosities and breakdown viscosities of the flours substituted with NTS, HTS and ATS were higher than those of the control, and these values of the flour with PTS were lower than those of the control. This shows that native, hydroxypropylated and acetylated tapioca starches swell and collapse easily during the heating process as compared with wheat starch, while phosphorylated cross-linked tapioca starch is more difficult to swell and disperse as compared with NTS, HTS, ATS and wheat starch.

Final viscosity of the flour substituted with HTS, ATS and PTS significantly decreased as compared with that of NTS and the control. This result suggests that gelatinized wheat flour gel containing hydroxypropylated, acetylated and phosphorylated cross-linked tapioca starches apparently retards gelling as compared with native tapioca starch. Setback of the flour substituted with PTS was higher than that of the flour with HTS. This result indicates that PTS gel has a more rigid network of amylose and/or amylopectin than HTS gel.

Gelatinization properties of substituted doughs.

Gelatinization properties of the doughs substituted with native and various modified tapioca starches are summarized in Table 3. Large endothermic peak (Peak 1) at around 55–100°C and small endothermic peak (Peak 2) above 100°C were observed. The former is ascribed to the gelatinization of starch and the latter is attributed to the melting of amylose-lipid complexes as mentioned by Kugimiya *et al.*[26] T_{o1} and T_{f1} of the doughs were not significantly different among the samples tested. T_{p1} of the dough prepared from the flour substituted with NTS, ATS and PTS sifted higher temperature as compared with the value of the control. As tapioca starch shows a higher gelatinization temperature than wheat starch on a DSC curve,[27] the dough prepared from the flour substituted with NTS, ATS and PTS might show higher T_{p1} on the DSC curve. The dough prepared from HTS showed lower T_{p1} among modified tapioca starches, and the temperature was similar to the control. This seems that degree of hydroxypropylation of 4–5% to tapioca starch significantly lowers the gelatinization temperature, and the gelatinization temperature becomes almost the same as the temperature of wheat starch. Endothermic enthalpy change (ΔH_1) of the dough with HTS, ATS and PTS was lower than

Table 3. Gelatinization characteristics of dough substituted with native or various modified tapioca starches for 20% of wheat flour.

Tapioca starch	Peak 1				Peak 2			
	T_{o1} (°C)	T_{p1} (°C)	T_{f1} (°C)	ΔH_1 (J/g)	T_{o2} (°C)	T_{p2} (°C)	T_{f2} (°C)	ΔH_2 (J/g)
Control	57.5 ± 2.0^a	72.4 ± 5.1^a	96.2 ± 3.3^a	3.5 ± 0.4^a	107.4 ± 4.0^b	114.5 ± 0.2^b	124.4 ± 3.8^b	1.8 ± 0.4^c
20% NTS	54.3 ± 1.9^a	85.4 ± 1.0^b	95.1 ± 1.1^a	4.4 ± 0.2^b	103.2 ± 1.9^a	112.7 ± 1.4^b	123.1 ± 0.4^b	1.4 ± 0.4^{abc}
20% HTS	55.1 ± 1.5^a	76.4 ± 6.6^a	91.0 ± 3.5^a	3.9 ± 0.5^a	101.0 ± 0.7^a	112.3 ± 1.5^b	119.0 ± 1.1^b	1.0 ± 0.4^{ab}
20% ATS	56.0 ± 1.9^a	84.8 ± 0.7^b	96.0 ± 3.1^a	4.1 ± 0.3^a	103.2 ± 1.4^a	113.5 ± 0.7^b	123.3 ± 0.7^b	1.3 ± 0.3^{abc}
20% PTS	57.3 ± 1.9^a	85.2 ± 0.2^b	96.8 ± 3.6^a	4.1 ± 0.3^a	101.2 ± 0.5^a	108.4 ± 3.8^a	119.6 ± 0.9^b	0.9 ± 0.1^a

T_o, onset temperature; T_p, peak temperature; T_f, final temperature ΔH, endothermic enthalpy change. Abbreviations are the same as in Table 1. Values are the average and standard deviation of triplicate. Numbers followed by different letters in the column are significantly different at $p<0.05$.

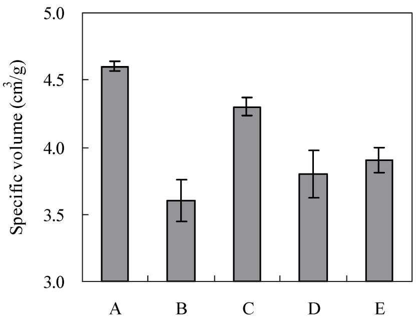

Fig. 2. Specific volume of bread prepared from the flour substituted with native or various modified tapioca starches for 20% of wheat flour.

A, control; B, 20% NTS; C, 20% HTS; D, 20% ATS; E, 20% PTS. Mean values±SD of triplicate.

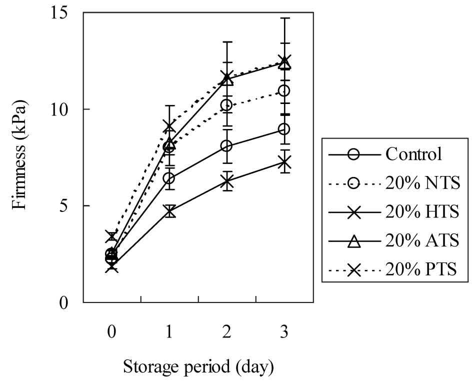

Fig. 3. Firmness of bread crumbs prepared with the flour substituted with native or various modified tapioca starches for 20% of wheat flour.

Each datum is a mean±SD of hexaplicate.

that with NTS. This suggests that each modified starch may be easier to change mainly its crystalline structure than native tapioca starch. ΔH_1s of the dough prepared from the flour substituted with 20% of HTS, ATS and PTS were the same as the value of the control. Moreover, gelatinization temperatures (T_{o1}, T_{p1} and T_{f1}) and endothermic enthalpy change (ΔH_1) of the dough prepared from the flour substituted with 20% (w/w) of HTS were quite similar to those values of the control.

Melting temperatures of amylose-lipid complexes (T_{o2}, T_{p2} and T_{f2}) of the doughs were not significantly different from each other except for T_{p2} of the dough prepared from the flour substituted with PTS. Eliasson[28] reported that the endothermic transition of the amylose-lipid complex was shifted towards higher temperatures, as the water content decreased. The dough prepared from the flour with PTS contained more water than that prepared from the flours with NTS or ATS (Table 1). Therefore, lower melting transition temperature may be attributed the higher water content of the dough.

Effect of various modified tapioca starches on loaf volume.

The substitution of native and modified tapioca starches to wheat flour decreased specific volume as compared with the control (Fig. 2). Although 8% (w/w) of vital gluten was substituted for wheat flour, this gluten might be insufficient to improve loaf volume. The bread made from the flour substituted with HTS had bigger volume than that prepared from the flour substituted with other tapioca starches. This difference could be attributed to water content in the dough and gelatinization properties of the tapioca starches. Because the dough prepared from the flour substituted with HTS showed a similar water absorption and gelatinization temperature or enthalpy change in starch gelatinization as compared with the control (Tables 1 and 3), gelatinization of both HTS and wheat starch in the dough prepared from the flour substituted with HTS might not disturb dough expansion during baking,[29] and show bigger volume than the breads from other flours.

Effect of various modified tapioca starches on crumb texture.

Bread crumbs prepared from the flour substituted with NTS, HTS and ATS had a sticky texture, while that from the flour with PTS had a dry texture. This difference would be related to peak viscosity on an Amylograph. The flour substituted with NTS, HTS and ATS showed higher peak viscosity, while that with PTS showed lower peak viscosity than the control (Table 2).

Firmness of the bread crumbs baked with the flour substituted with native and various modified tapioca starches is shown in Fig. 3. Bread crumb from the flour with HTS was the softest and retarded staling as compared with the control after three days of storage. Although the firmness of bread crumbs prepared from the flour with NTS and ATS was similar to the value of the control at baked day (day-0), bread prepared from the flour with NTS and ATS increased in hardness after one day of storage. Bread prepared from the flour with PTS had significantly hard crumbs at baked day (day-0). Takasaki *et al*.[15] reported that substitution of 20% hydroxypropylated cross-linked potato starch to wheat flour had no effect on firmness of bread crumbs as compared with the control, and with the crumbs made softer than the crumbs from the flour with native potato starch. Inagaki and Seib[12] and Toufeili *et al.*[13] reported that bread with cross-linked barley starch accelerated bread firming during storage. Although we used chemically modified tapioca starch, the results that hydroxypropylated starch retarded bread staling and cross-linked starch accelerated staling were very similar to previous results.[12,13] In this study, we revealed that acetylated tapioca starch accelerated bread staling as well as cross-linked one. Several workers reported that higher amount (>20% (w/w)) of tapioca starch substitution deteriorates bread qualities.[24,25,30,31] However, our results suggested that HTS is expected to apply to breadmaking as an improver, since it possesses a prominent anti-staling effect.

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ヒドロキシプロピル化，アセチル化および燐酸架橋タピオカ澱粉を置換した小麦粉の糊化特性および製パン性

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ヒドロキシプロピル化タピオカ澱粉（HTS），アセチル化タピオカ澱粉（ATS）および燐酸架橋化タピオカ澱粉（PTS）の3種類の化学修飾タピオカ澱粉で20%（w/w）置換した小麦粉の糊化特性と製パン性を，未処理タピオカ澱粉（NTS）で置換した小麦粉，ならびにタピオカ澱粉で置換していない小麦粉（対照）の場合と比較した．DSCによるドウの糊化温度および糊化吸熱エンタルピーを測定したところ，HTSを20%含むドウは対照のドウと類似していた．NTS，HTS，ATSは小麦澱粉よりも膨潤，破壊しやすかったが，PTSはNTS，HTS，ATSおよび小麦澱粉よりも膨潤しにくく，澱粉粒は崩壊しにくかった．ファリノグラフによりドウのミキシング特性を測定したところ，HTSを除くタピオカ澱粉置換粉の吸水率は対照よりも減少した．いずれの置換粉もミキシング時間は対照より短くなったが，ミキシング耐性は同等であった．置換粉を用いて焼成したパンの比容積は顕著に減少したものの，HTSを用いたものは置換粉の中では最も大きな比容積を示した．ATS，PTS置換粉のパンのクラムはNTS置換および対照よりも保存中に硬くなったが，HTS置換粉では柔らかであった．HTS，ATS，PTSの3種類の化学修飾タピオカ澱粉の中ではHTSが最も小麦粉置換物として製パンに適していることが明らかとなった．