Modification of Starch by Dry Heating with Ionic Gums

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ABSTRACT

Waxy maize (native and hydroxypropylated [HP]) and potato starches were impregnated with ionic gums (sodium alginate, CMC, and xanthan, 1% based on starch solids) and heat-treated in a dry state for 0, 2, or 4 hr at 130°C. Effects of the dry heating on paste viscosity (RVA) and clarity (light transmittance) were examined. Heat treatment with sodium alginate and CMC raised the paste viscosities of native and HP waxy maize starches, but decreased that of potato starch. Xanthan provided the most substantial changes in paste viscosity among the tested gums. It appeared to heavily restrict granule swelling of the waxy maize starches, but it increased swelling of potato starch granules. Dry heating raised the paste viscosity of all the starch-gum mixtures tested, except the potato starch-alginate mixture. The final viscosity at 50°C of a 7% paste was raised in all other starches by ≈500–1,000 cP by this treatment. The paste of waxy maize starch-gum products became opaque and shorter textured by the heat treatment, regardless of the gum type, whereas potato starch-gum products did not show any obvious change in paste clarity. Ionic gums could behave as cross-linking agents as well as form graft copolymers through heat-induced ester formation. This simple heating process with ionic gums could be used as a modification method for starch.

Heat-moisture treatment has been extensively studied as a physical method for starch modification, but heat treatment of starch under dry conditions has been studied only rarely. One exception is pyrodextrin production by thermal degradation of starch at high temperatures (Greenwood 1967).

Heating to lower temperatures has other effects. In 1972, Goto reported that some physical properties of starch could be changed during thermal dehydration in an electric oven. Starches of various origins heated at 80–120°C for ≤20 hr exhibited reduced maximum viscosity and pasting temperatures. Seguchi et al (1984, 1988) found that the hydrophobic nature of wheat starch was enhanced by heating it at 120°C for several hours, a change favorable for cake baking. The dry heating process as an alternative to chemical modification for starches or flours of various origins has been patented (Chiu et al 1998, 1999). Claimed is the heating of starch or flour at >100°C, preferably for several hours, to yield functionality equivalent to that obtained by chemical cross-linking. Dehydration (<1% moisture) before heating and alkalinity (pH 8–9.5) of starch enhanced the effect of heating. Starches treated by this process produced pastes with increased resistance to viscosity breakdown and a noncohesive or "short" texture.

Reactive compounds can be added to produce chemical modifications during the heat treatment. As an example, a small amount of levoglucosan added to starch before heating gave rise to enzyme-resistant glycosidic linkages through heat-induced reaction with the hydroxyl groups of starch (Theander and Westerlund 1987; Siljestrom et al 1989).

Natural or modified gums (hydrocolloids), often used together with starch in various food products, can modify the characteristics of the end products (Christanson et al 1981; Alloncle et al 1989; Sudhakar et al 1992; Ferrero et al 1993a,b; Ferrero et al 1994; Rayment et al 1995; Sekine 1996; Liu and Eskin 1999). Instead of the simple aqueous mixing of starch and gums, processes accompanied by heating such as jet-cooking (Fanta and Christianson 1996) and extrusion (Miladinov and Hanna 1995, 1996; Dudacek et al 1999) have been used to provide new functionality to mixture products. But no one heretofore has done dry heat treatments of starch in the presence of gums.

In this study, waxy maize (unmodified and hydroxypropylated [HP]) and potato starches were heat-treated in a dry state after being impregnated with a gum (sodium alginate, sodium carboxymethylcellulose, or xanthan). The effect of the heat treatment on the pasting behavior of the starches was examined.

MATERIALS AND METHODS

Starches and Gums

Waxy maize and potato starches were obtained from A. E. Staley Mfg. (Decatur, IL) and Penford Food Ingredients (Englewood, CO), respectively. Food-grade xanthan gum and sodium alginate were obtained from NutraSweet (Chicago, IL). Carboxymethylcellulose was a low-viscosity type (7LF) from Hercules (Wilmington, DE).

Waxy maize starch was partially hydroxypropylated (Tsuzuki 1968). Starch (300 g, db) was reacted with propylene oxide (23 mL), with an alkaline catalyst (40 mL, 7% NaOH), and sodium sulfate (43 g) by stirring in an aqueous medium (500 mL) for 24 hr at 45°C.

Sodium alginate, CMC, or xanthan (0.4 g) was slowly added to distilled water (70 mL) with vigorous stirring. Starch (39.6 g, db) was added to the gum solution, and the dispersion was stirred for 30 min at room temperature. The whole dispersion was transferred into a glass dish and dried at 45°C in an oven to a moisture content of <10%, then ground into a powder. The gum was added at a concentration of 1.0%, based on starch.

Heat Treatment

The starch-gum mixture was heated in an aluminum dish in an electric oven at 130°C for 2 or 4 hr. The starch itself was concurrently heat treated without gum under identical conditions.

Paste Viscosity

Paste viscosity of samples was measured using a Rapid Visco Analyser (RVA) (Newport Scientific Inst., Australia) at a concentration of 7.0% (1.96 g in 28 g total). The standard procedure (No. 1) provided by the RVA manufacturer (starting and ending at 50°C, 3 min holding at 95°C, 15 min total analysis time) was used.

Light Transmittance of Paste

Starch, starch-gum mixture, or starch-gum product dispersion (1% in water, w/w) was pasted in a sealed bottle by heating for 30 min in a boiling water bath with continuous stirring. The paste was cooled at room temperature for 1 hr. Transmittance was measured at 650 nm.

RESULTS

Heat Treatments Without Gum

Unmodified waxy maize starch displayed a trend in which the paste viscosity gradually decreased as the heating time increased, but the overall viscogram pattern was not significantly changed (Fig. 1). However, in HP waxy maize starch, no significant change in pasting viscosity or pattern was observed. Potato starch showed

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changes in viscograms that were different from those of waxy maize starch. Using the same heat treatment, peak viscosity underwent noticeable reduction, but the final viscosity was not changed. Similar results have been reported (Goto 1972; Chiu et al 1998, 1999). This change could result from either restricted granular swelling by the heat treatment or greater granule disruption during the early stage of pasting. Despite the differences in viscograms, one feature commonly observed with all three heat-treated starches was a slight apparent pasting temperature decrease.

Each starch was dispersed in water (1:4 starch-to-water), and the pH of the dispersion was measured. The commercial waxy maize starch was acidic (pH 5.1), whereas dispersions of potato starch and HP waxy maize starch had values of pH 7.4 and 7.0, respectively. To remove any effect of acidity, an aqueous dispersion of waxy maize starch was neutralized with 0.1*M* Na_2CO_3, followed by washing. The starch was recovered by filtration and dried. As shown in Fig. 2, the neutralized waxy maize starch was resistant to the heat treatment (130°C, 4 hr). This confirmed that the acidity of the commercial waxy maize starch was responsible for the viscosity decrease brought about by the dry heat treatment. The slight alkalinity of the commercial potato starch was also removed by neutralization with 0.2*M* HCl, followed by washing. The neutralized starch had the same viscogram patterns as the native starch (data not shown). Therefore, unlike native or modified waxy maize starch, potato starch is inherently sensitive to dry heat treatment.

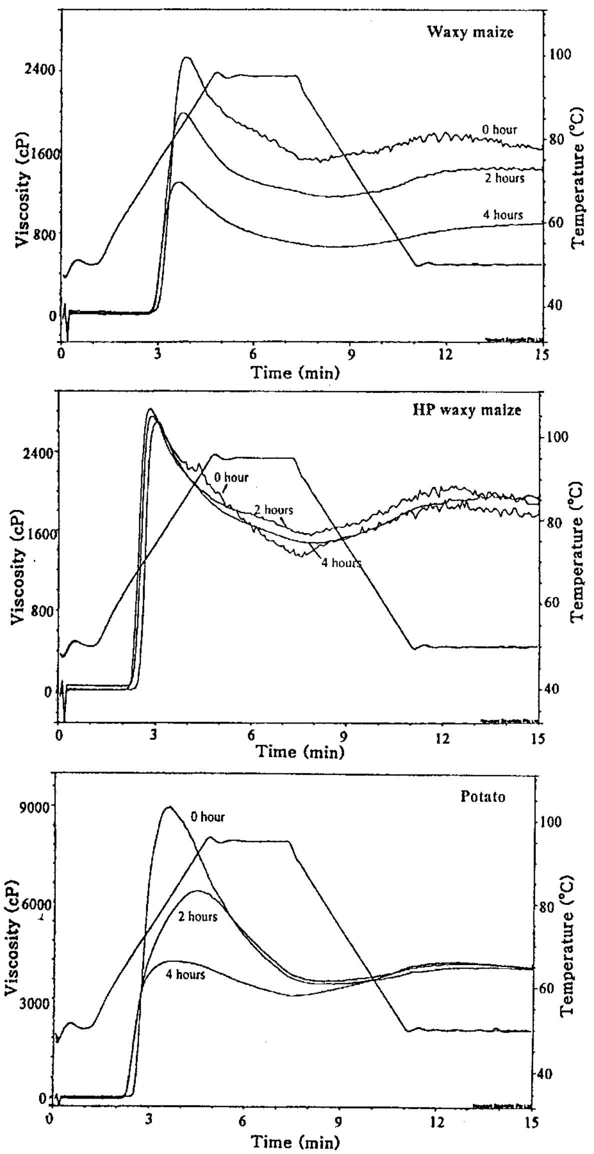

Fig. 1. Paste viscograms of native waxy maize, hydroxypropylated (HP) waxy maize, and potato starches heat treated at 130°C for 2 or 4 hr.

Goto (1972) compared potato and maize starches and found that potato starch was more sensitive to dry heating (80–120°C for ≤20 hr), as indicated by pasting properties. Sekine et al (2000) found that B-type starches were more sensitive than A-type starches to a heat dehydration process (120°C, 2 hr). The B-type starches showed greater changes in thermal and rheological properties. In differential scanning calorimetry, heat-treated B-type starches gelatinized at a lower temperature with less enthalpy, indicating reduced thermal stability of starch granules, but without any clear change in X-ray diffractograms. So, any structural changes induced by dry heating may occur in the amorphous regions of granules. This change is more prevalent in B-type granules.

Heat Treatment with Gum

Incorporating ionic gums slightly changed the pH of the starch. The pH of a native waxy maize starch slurry (pH 5.1) was raised to 5.6–6.5, depending on the gum incorporated. The pH of a slurry of potato starch was reduced slightly to pH 6.7–7.2 by gum addition. It was assumed that no or negligible acid-catalyzed decomposition occurred in starch-gum mixtures during heat treatment.

The pasting viscosity profile in Fig. 3 shows the effect of heating the three starches with sodium alginate. Sodium alginate without heat treatment did not produce any significant change in the viscogram of waxy maize starch (0 hr, Figs. 2 and 3). But dry heating (130°C, 2 or 4 hr) raised the viscosity throughout the RVA analysis. After 2 hr at 130°C, the peak and final viscosity values increased by ≈900 and 500 cP, respectively. The viscosity increase continued as the heating period increased from 2 to 4 hr, but the increment was less than that occurring within the initial 2 hr. Heat treatment alone lowered the pasting temperature (Fig. 1), but the decrease was more significant with the gum. The overall viscogram revealed that waxy maize starch granules with sodium alginate became more readily hydrated and swollen after heat treatment.

HP waxy maize starch displayed a trend similar to that of unmodified starch (Fig. 3), but different from that of native waxy maize starch, where the first 2 hr of heating produced a much smaller increase in final viscosity than did the next 2 hr. Both breakdown (viscosity decrease in the hot stage) and setback (viscosity increase during cooling) became greater upon heat treatment, resulting in a noticeable increase in final viscosity (≈1,000 cP).

Potato starch behavior was substantially influenced by incorporating sodium alginate, even when the gum was mixed with the starch without heating (0 hr, Fig. 3), the paste viscogram became totally different (Shi and BeMiller 2002). Dry heating of potato starch without gum also reduced peak viscosity, breakdown, and setback (Fig. 1). Because of its inherent phosphate ester groups, potato starch is sensitive to both organic and inorganic ions (Muhrbeck and Eliasson 1987; Kelly et al 1995; Shi and BeMiller 2002). Peak viscosity suppression is a usual consequence of adding ionic

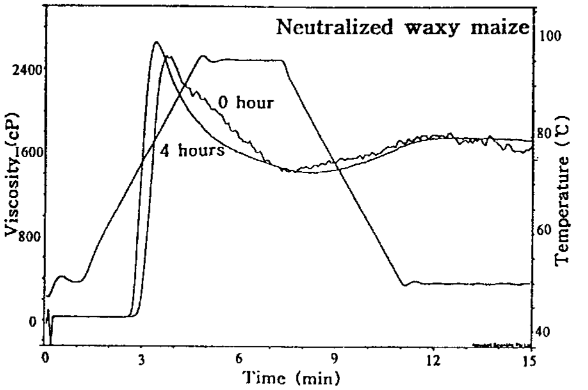

Fig. 2. Paste viscograms of neutralized waxy maize starch before and after dry heat treatment at 130°C for 4 hr.

compounds to potato starch (Shi and BeMiller 2002), an effect often explained as charge repulsion between ions. Therefore, we hypothesize that ions from the incorporated sodium alginate suppressed peak viscosity development of potato starch by restricting granular swelling (Shi and BeMiller 2002).

Dry heating further decreased paste viscosity of the potato starch-alginate mixture. The viscosity decrease appeared throughout the viscogram temperature profile, a trend opposite to that for native and HP waxy maize starch. The heat-induced restriction of granular swelling, displayed only by potato starch without gum (Fig. 1), probably also occurred with sodium alginate. In addition, due to ionic repulsion between the carboxylate groups of sodium alginate and the phosphate groups of potato starch, there might have been a reduction in associated interactions between alginate and potato starch of the type believed to be responsible for the viscosity changes in waxy maize starch pastes. Therefore, restricted swelling of potato starch granules might be the sole reason for the viscogram change induced by heating, with formation of distarch phosphate cross-linkages being primarily responsible.

Incorporation of sodium carboxymethylcellulose (CMC, low-viscosity type), followed by heat treatment, increased the paste viscosity of native and HP waxy maize starches, whereas potato starch displayed mixed results (Fig. 4). Heat treatment of unmodified waxy maize starch produced a paste more resistant to breakdown, the shear stability significantly increased final viscosity. The waxy maize starch-CMC mixture heated for 4 hr produced a final viscosity of ≈2,500 cP, almost 1,000 cP higher than that of the unheated mixture.

For HP waxy maize starch, however, such heat treatment mainly increased the peak viscosity (Fig. 4). Unlike native waxy maize starch, the hot HP starch-CMC paste was susceptible to breakdown. As a result, dry heating alone increased final viscosity by <500 cP. HP starch granules swell more readily in water than native starch granules. Therefore, upon mixing with an aqueous gum solution, the modified starch might be more accessible to the ionic gum molecules (more impregnated). In both native and HP starches, the paste viscosity profile was not significantly changed after 2 hr of heating with CMC.

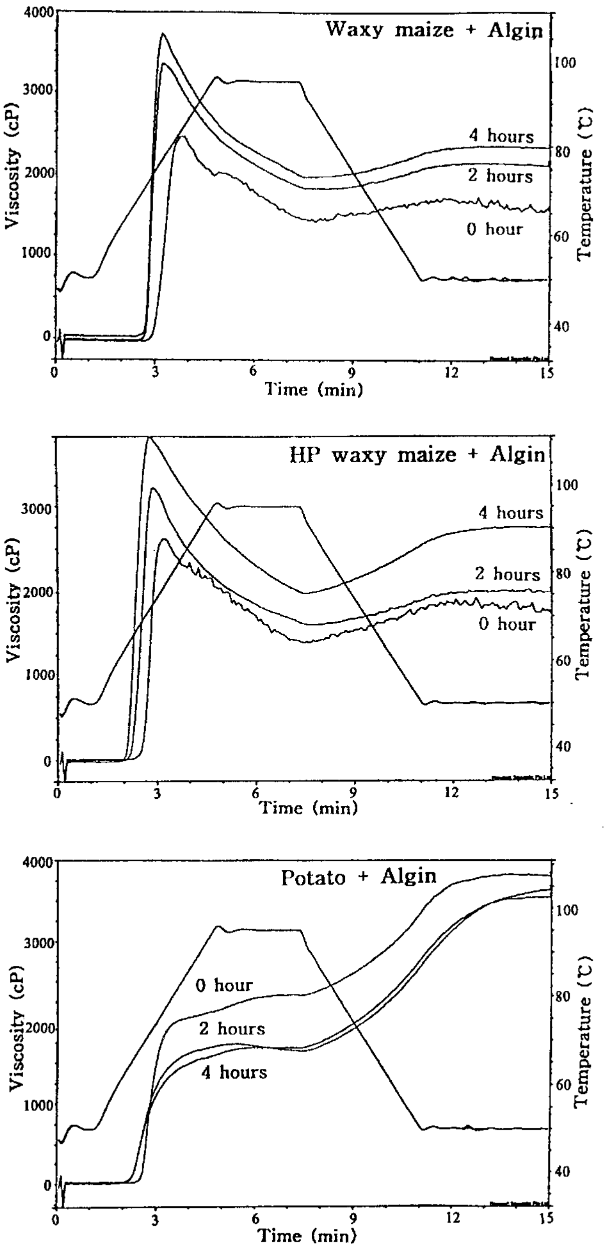

Fig. 3. Paste viscograms of native waxy maize, hydroxypropylated (HP) waxy maize, and potato starches heat treated at 130°C for 2 or 4 hr with sodium alginate (Algin, 1% based on total solids).

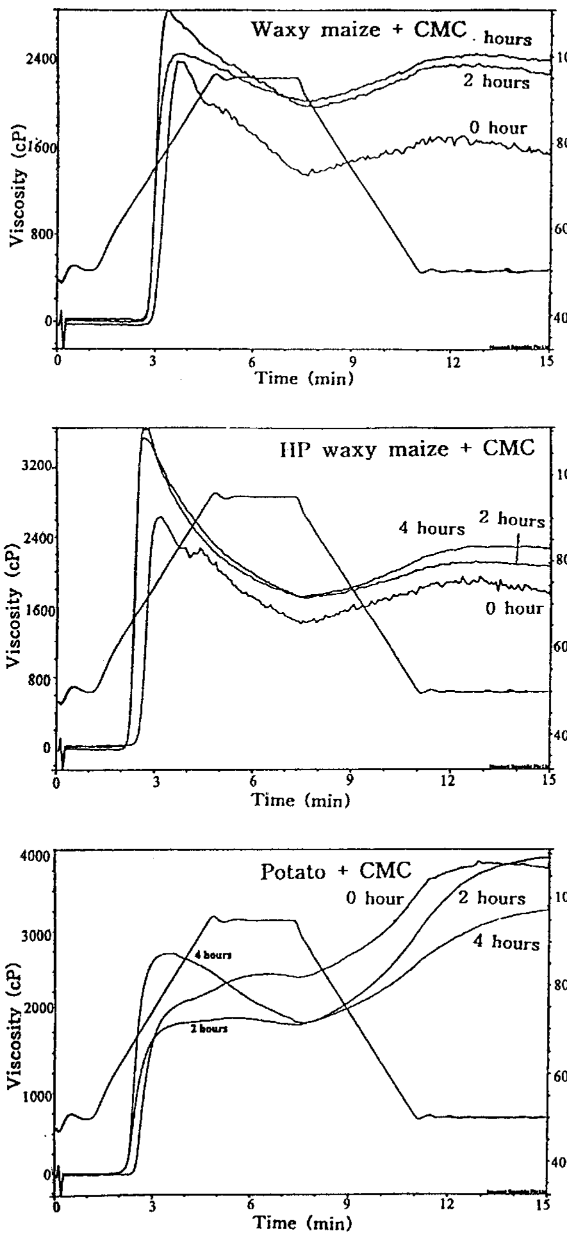

Fig. 4. Paste viscograms of native waxy maize, hydroxypropylated (HP) waxy maize, and potato starches heat treated at 130°C for 2 or 4 hr with sodium carboxymethylcellulose (CMC, 1% based on total solids).

As observed with sodium alginate, CMC without heat treatment inhibited the swelling of potato starch granules (0 hr in Fig. 4). By heating the starch-CMC mixture for 2 hr, the inhibitory effect appeared to increase and, thus, the starch paste reached a plateau earlier (at 75°C). But the setback was greater than that of a unheated control, resulting in similar values for final viscosity. Upon heating for 4 hr, however, the starch-CMC mixture gave a peak viscosity of ≈500 cP higher than that of the sample heated for 2 hr, but the intermediate breakdown and low setback gave a final viscosity lower than that of the sample heated for 2 hr. When CMC was present, the effect of heating on the pasting viscosity profile of potato starch was different from that observed with sodium alginate. Also, significant differences in viscogram patterns were produced by the two different heating periods (2 and 4 hr). It is apparent that the effect of this anionic gum, both with and without dry heating with potato starch, is rather different from that produced by sodium alginate.

Among the ionic gums examined, xanthan rendered the most profound effect on the paste viscosity of the heat-treated starches (Fig. 5). Upon dry heating with xanthan, waxy maize starch became resistant to the pasting process. No peak viscosity was seen, and granule swelling appeared to be highly retarded, even through the 95°C holding period. The final viscosity was higher than that of the unheated counterpart.

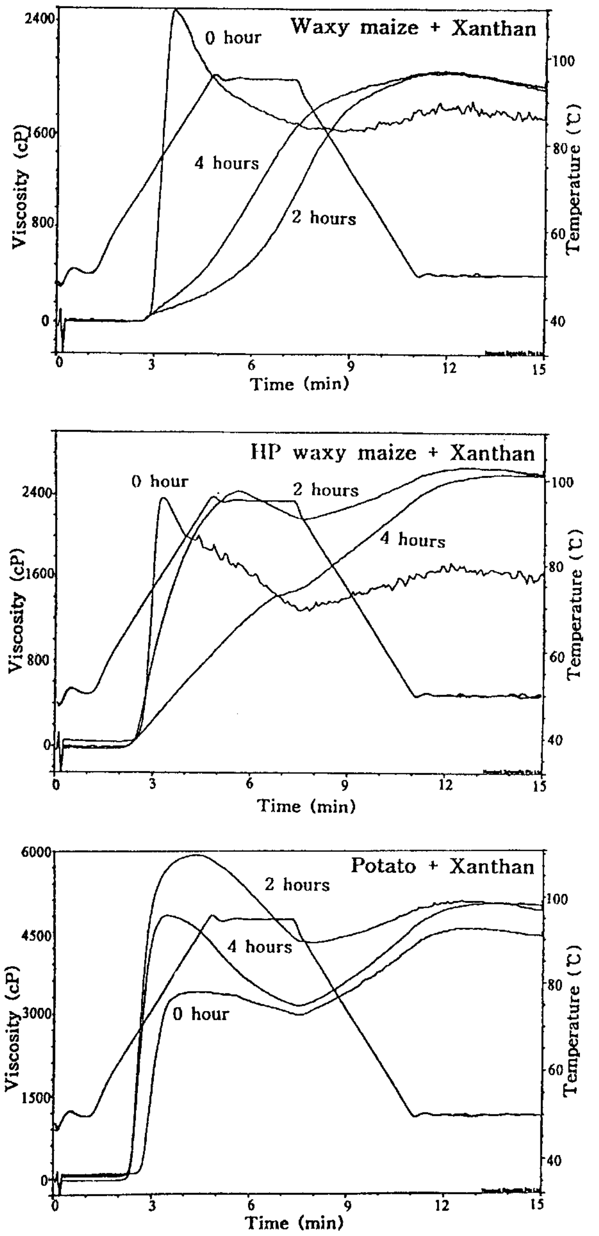

Fig. 5. Paste viscograms of native waxy maize, hydroxypropylated (HP) waxy maize, and potato starches heat treated at 130°C for 2 or 4 hr with xanthan (1% based on total solids).

Similar trends were found with HP waxy maize starch, but the change appeared to be more gradual (Fig. 5). The viscosity peak of the starch heated for 2 hr occurred after the temperature had reached 95°C, and the resulting shortened time of the 95°C hold resulted in minimal breakdown. Extending the heating to 4 hr resulted in a substantial restriction in granular swelling as evidenced by the viscosity increase becoming almost linear with continuous viscosity development from the pasting temperature to the final temperature. The final viscosities of the heated samples at 2 and 4 hr were similar, both values being ≈1,000 cP higher than that obtained with the unheated sample.

As with alginate and CMC, the swelling of potato starch granules was affected by xanthan addition (Fig. 5), but the potato starch-xanthan mixture displayed a pasting profile different from that observed with other gums (Figs. 3 and 4). The potato starch-xanthan mixture subjected to 2 hr of heating produced a paste with a peak viscosity about double of that of the unheated mixture. Heating for 4 hr decreased the peak viscosity from that reached by the product of 2 hr of heating, but the peak viscosity was still greater than that achieved with the unheated mixture. Despite the significant difference in peak viscosity, the final viscosity values of the heat-treated samples at different periods were the same, and ≈500 cP greater than that of the unheated sample.

Dry heat treatment with ionic gums raised the paste viscosity of waxy maize and HP waxy maize starches. Potato starch gave mixed results. The viscosity increase suggests that the gum molecules permanently modified the starch during dry heating.

Paste Clarity

Table I shows the transmittance of dilute pastes (1% solids) of heat-treated starch-gum mixtures. In the absence of gum, commercial waxy maize starch displayed an increase of paste transmittance as the heating time increased. As indicated earlier, acid-catalyzed degradation or transglycosidation was responsible for the increase in paste clarity. Transmittance of the unmodified waxy maize starch paste became stable to the heat treatment after neutralization (data not shown), confirming this hypothesis. HP waxy maize starch and potato starch formed pastes that were clearer than that of native waxy maize starch. Transmittance for those starch pastes was not changed by the heat treatment because of the near neutrality of the starches. It had been expected that heated potato starch would show reduced paste clarity because of restricted swelling (Fig. 1), but no significant change in paste clarity was found.

TABLE I
Pasting Viscosity Profile of Dry Heat Treatment (130°C) of Starch-Gum Mixtures

	Transmittance of Dilute Pastes (1% solids)		
Sample	**0 hr**	**2 hr**	**4 hr**
Waxy maize starch			
No gum added[a]	49.5	56.7	62.1
Sodium alginate	44.7	22.7	19.9
CMC	41.9	18.1	11.1
Xanthan	44.3	21.2	21.3
HP waxy maize starch			
No gum added	55.3	57.3	53.1
Sodium alginate	51.6	31.8	21.8
CMC	47.0	28.8	27.6
Xanthan	47.9	34.2	25.6
Potato starch			
No gum added	86.3	87.5	87.4
Sodium alginate	87.2	81.0	85.7
CMC	87.5	88.6	76.1
Xanthan	90.0	92.9	89.9

[a] Unneutralized, commercial waxy maize starch (slurry pH 5.1).

When gums were present, heat treatment reduced the clarity of native and HP waxy maize starch pastes, whereas no clear trend was observed for potato starch pastes. All three gums exhibited the same trend of decreasing transmittance in proportion to heating time, although the effects of heating on viscograms were different for the three gums used (Figs. 3–5).

DISCUSSION

Commercial potato starch exhibited a viscogram change by dry heat treatment alone, whereas native and HP waxy maize starch at neutral pH did not. We first suspected that there might have been a heat-moisture effect at the early stage of heating when residual moisture was not fully removed. The higher susceptibility of potato starch appeared explainable because potato starch normally contains more moisture than waxy maize starch. In addition, even at equal moisture contents, potato starch is more affected by heat-moisture treatment than cereal starches are (Hoover and Vasanthan 1994). But it has also been reported that starch was changed in pasting characteristics by heat treatment, with drier starch being more affected (Chiu et al 1999). In a separate experiment, we washed the potato starch with acetone and dried the starch at 45°C to a moisture content of <4%. This starch was heat treated for comparison. In agreement with Chiu et al (1999), we observed an increased effect of heating on the paste viscogram as the moisture content of starch was reduced (data not shown, but similar to Fig. 1). The starch might loose most residual moisture during heating for 2 hr at 130°C.

Because no change in X-ray diffractograms was observed after dry heat treatment (Sekine et al 2000), any possible structural changes were expected only in the amorphous regions of starch granules. There might be a difference in the heat susceptibility of the amorphous regions between B- and A-type starches, which might be responsible for the heat-susceptibility difference between starches of different origins, but it was hypothesized that any structural change would require at least minimal presence of moisture or another plasticizer for the starch chains to be mobile. A more likely explanation for the heat-induced change in potato starch could be a reaction between phosphate ester groups and hydroxyl groups in adjacent glucosyl units, forming a diester linkage (cross-linking) between starch chains. Experimental results that moisture reduction enhanced heat-induced changes in the viscogram support this theory. To confirm the reaction, precise chemical analyses will be required.

A variety of neutral or less ionic gums, including guar gum, methylcellulose, hydroxypropylmethylcellulose, and gum arabic were also tested, but unlike the highly ionic gums tested (xanthan gum, sodium alginate, and CMC), all of which were effective, the gums with no or slight anionic charges did not change the paste viscosity of the dry heat-treated starches. As potato starch appeared to become cross-linked (through distarch phosphate ester formation) when the starch alone was dry heated, similar ester formation could occur when starch and ionic gum mixtures are dry heated, but each anionic gum molecule is multicarboxylated so that more than one starch chain is likely to react with a gum molecule. The number of starch chains linked to a gum molecule would be important in determining the pasting behavior. If there are two or more starch chains connected to a gum molecule, the gum acts as a cross-linking agent. But if one starch polymer molecule is ester-linked with one gum polymer molecule, the product becomes a starch-gum grafted copolymer. The overall physical property of the copolymer would be dependent on the nature of the gum grafted and the position of the graft on each polymer. The mixed results in the paste viscograms could arise from the two possible reactions occurring in different ratios, leading to different behaviors. Based on paste viscosity and clarity data, we concluded that heat-induced ester formation between starch and ionic gums was more prevalent in waxy maize starch than in potato starch, repulsion by the phosphate groups in potato starch possibly being the cause of the low reactivity.

Precedent for esterification comes from the facts that common corn starch reacted with succinic anhydride in anhydrous pyridine is highly cross-linked (Fannon and BeMiller 1992) and that simply dropping the pH to acid values before recovering and drying, when making carboxymethylated starch, imparts characteristics of cross-linking. The results obtained with the waxy maize starch control could be due to presence of protein (as a source of carboxyl groups) throughout the granule, although the amount of protein in waxy maize starch is much lower than it is in common corn starch (Han and Hamaker, *unpublished*).

Among the gums used, xanthan displayed a greater tendency to form multistarch esters. A paste of waxy maize starch heat treated (130°C, 4 hr) in the presence of xanthan, immediately after holding (RVA) at 95°C for 3 min was examined by light microscopy. The treated waxy maize starch retained its granular form with only minor swelling, whereas pastes made from unheated starch-xanthan mixtures revealed almost complete rupture of the granules (results not shown). With potato starch treated with xanthan, the pasted granules, although highly swollen, were more intact and less ruptured compared with those of untreated starch (results not shown). Thus, we hypothesize that granule stability is produced by multiester linkages as speculated with waxy maize starch, that the degree of esterification in potato starch was less than that in waxy maize starch, based on the differences in the swelling, and that cross-linking allowed the paste viscosity of potato starch to continue increasing during the early heating stage (Fig. 5).

The esterification reaction between starch and ionic gums can be influenced by various reaction parameters such as pH, temperature, and presence of a catalyst. Future research on those parameters to control the reaction to create the desirable pasting characteristics for utilization of the starch-gum reaction product will be done.

CONCLUSIONS

Neutral waxy maize starch was relatively resistant to dry heat treatment, whereas potato starch was susceptible. It was hypothesized that the phosphate groups of potato starch formed diester cross-links during the dry heating. Likewise, incorporation of an ionic gum in starch, followed by dry heating, could result in ester formation between starch and gum. But potato starch was less reactive than waxy maize starch, possibly due to ionic repulsion between the like charge of starch and gum. The heat-induced reaction raised paste viscosity but decreased paste clarity. Changes in starch behavior seemed to be a function of the type of gum. With proper selection of ionic gum and control of reaction conditions, a dry heating process can provide desirable starch functionality. This process is simple and efficient, and produces no byproducts.

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