

Effects of food gums on viscosities of starch suspensions during pasting

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Abstract

Pasting curves of starches in gum (hydrocolloid) solutions at low concentrations (starch 3.6%, gum 0.4%) were produced with a Brookfield viscometer. Gums produced a variety of effects on viscosities of starches during starch pasting (increase or decrease greatly or slightly or no effect). A viscosity increase before the normal starch pasting temperature was detected for normal maize starch in the presence of carboxymethylcellulose (CMC), gellan, xanthan, guar gum, and sodium alginate. Waxy maize, waxy rice, tapioca, regular rice, potato, and wheat starches gave mixed responses. It appeared that interactions between certain leached molecules, primarily amyloses, and certain gums were responsible for the viscosity increase occurring before starch pasting.

The pasting peak viscosity of potato starch was greatly decreased by negatively charged gums (CMC, carageenans, alginate, xanthan). The repelling forces between the phosphate groups on potato starch and the negative charges on molecules of these gums were hypothesized to be the cause. This hypothesis was supported by results from similar systems (potato starch plus salt solution and phosphorylated normal maize starch plus anionic gums), both of which systems also produced lower peak viscosities. It was found, by microscopic examination of potato starch heated to 95 °C in solutions of ionic gums without shear, that the gums altered the granule pasting process. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Starches; Gums; Hydrocolloids; Viscosity

1. Introduction

Starches and gums (hydrocolloids) are often used together in food systems to provide proper texture, control moisture and water mobility, improve overall product quality and/or stability, reduce costs, and/or facilitate processing. It is, therefore, important to understand interactions between starches and food gums that are critical to the functionalities they impart to food products. Results of previous experiments conducted to this end have been reviewed (Appelqvist & Debet, 1997).

Changes in apparent paste viscosity have been used to study interactions between starches and gums. When the temperature of a slurry of wheat starch in solutions of sodium alginate, xanthan, sodium carboxymethylcellulose, and guar gum was raised, it appeared that the gums lowered the gelatinization temperature of the granules because a measurable viscosity increase (Brabender ViscoAmylograph) occurred at a lower temperature as compared to the same wheat starch slurry heated without added gum (Christianson, 1982; Christianson, Hodge, Osborne, & Detroy, 1981; Christianson, Navickis, Bagley, & Wolf, 1984; Cross-

land & Favor, 1948). These results are congruent with those of Bean and Yamazaki (1978), who correlated the early-stage viscosity increase with an increase in granule diameter. Christianson et al. (1981, 1982, 1984) attributed the early onset of initial viscosity to the first-stage of granule swelling and believed that it was a function only of media viscosity, rather than of granule swelling. The solution obtained after removal of granules displayed stable, synergistic viscosity, which led them to suggest interactions between gum molecules and solubilized amylose and low-molecular-weight amylopectin molecules as a possible cause of the increase in paste viscosity.

Alloncle and Doublier (1990) found that, while G' increased, elasticity decreased when xanthan, guar gum, or locust bean gum were present in a normal maize starch paste. Liu and Lelievre (1992) reported that the presence of rice starch in a gellan or gellan-locust bean gum gel resulted in increases in G' with increases in temperature. The largest increase occurred as granules gelatinized, indicating that starch polymer molecules and/or granule fragments reinforced the gel in some way. Stress and strain at failure were reduced, but increased with increased amounts of gelatinized starch.

Others (Alloncle, Lefebvre, Llamas, & Doublier, 1989; Bahanassey & Breene, 1994; Ferrero, Martino, & Zaritzky,

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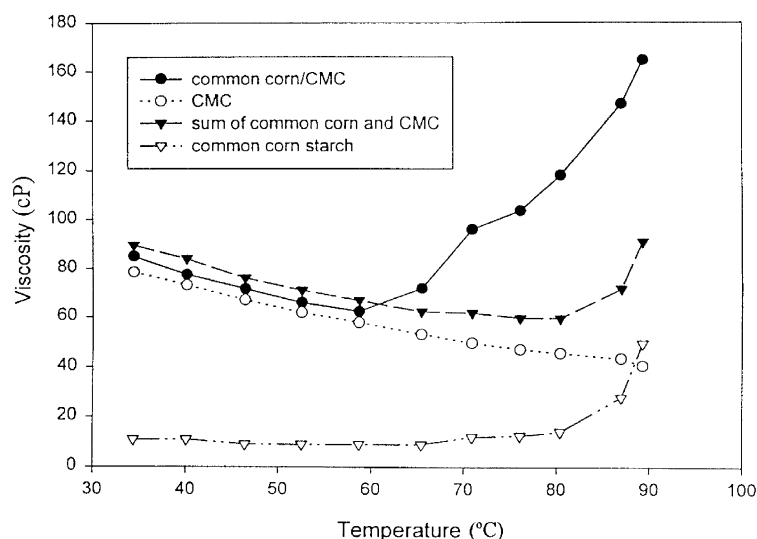


Fig. 1. Brookfield viscosities in centipoises (mPa s) of a suspension of normal maize (common corn) starch granules (3.6%) in water and in a CMC solution (0.4%) and the CMC solution alone heated from 35 to 90 °C, plus the sum of the viscosities of the gum solution and starch suspension.

1993a,b; Ferrero, Martino, & Zaritzky, 1994; Fonteneau & Germon, 1986; Liu & Eskin, 1998; Rojas, Rosell, & Benedito de Barber, 1999; Sekine, 1996) also observed synergistic interactions between a starch and a gum, but such effects were not always indicated (Appelqvist & Debet, 1997; Tye, 1988), leading to the hypothesis that the synergistic effect was due to phase separation (Alloncle et al., 1989; Alloncle & Doublier, 1991; Annable, Fitton, Harris, Phillips, & Williams, 1994; Appelqvist, Brown, Goff, Lane, & Norton, 1996; Kalichevsky & Ring, 1987) rather than to intermolecular associations. Promotion of intergranular association was also proposed (Abdulmola, Hember, Richardson, & Morris, 1996).

In most of the investigations, a granular starch and a gum powder were heated together in a Brabender Visco-Amylograph or rapid visco-analyzer (RVA). Many revealed an increase in viscosity at temperatures below the normal pasting temperature and gave the appearance of one or more gums lowering the pasting temperature (erroneously called gelatinization temperature) of some starches, and that is how the results were usually interpreted (Sudhakar, 1995). However, because of the instruments used and the experimental designs, it was not evident whether viscosity increases were due to changes in granule gelatinization and/or pasting, to gum solubilization, or to starch–gum interactions. And neither the Brabender Visco-Amylograph nor the RVA measures low viscosities. An objective of this research was to produce pasting curves at low concentrations of starch and gums (when changes in shape and size of starch granules may be less of a factor in any viscosity increase and interactions between starch granules and/or molecules and gums a stronger factor) using a traditional heating program. To do this required investigating changes in viscosity occurring below the threshold levels of the traditional instruments.

2. Materials and methods

2.1. Materials

Normal and waxy maize starches were obtained from A.E. Staley Mfg. Co. (Decatur, IL). Regular rice and waxy rice starches were obtained from California Natural Products (Lathrop, CA). All gums were obtained from commercial sources of food gums: carboxymethylcellulose (CMC) was from Hercules, Inc., type 7H3SF; hydroxypropylmethylcellulose (HPMC) was from Dow Chemical Company, type E 50.

2.2. Methods

Gum (1.8 g dry wt, 0.4%) was dissolved in 450 ml of dd water at room temperature with mechanical stirring in a C.W. Brabender Instruments Visco-Amylograph (South Hackensack, NJ) heating container. (When the gum was gellan, a solution was prepared by heating a 0.4% suspension of gellan to 95 °C. The gum solution was then cooled before adding starch.) Starch (16.2 g dry wt, 3.6%) was dispersed in the gum solution or water (as control). The mixture was heated using the Visco-Amylograph heating profile (1.5 °C/min) with a 2 min stop every 3 or 5 min to measure viscosity (3 min in transition regions). Viscosity was measured (in triplicate) in the heating container with a Brookfield Engineering Laboratories (Stoughton, MA) Viscometer (Programmable DV-II +) using spindle #5 at a shear rate of 200 rpm. The spindle was rotated in the medium for 1 min prior to a reading for temperature equilibration.

RVA analysis was done using standard profile I of Model RVA-4 (Newport Scientific, Warriewood, Australia). Starch (2.5 g, dry wt) was suspended in dd water or gum solution

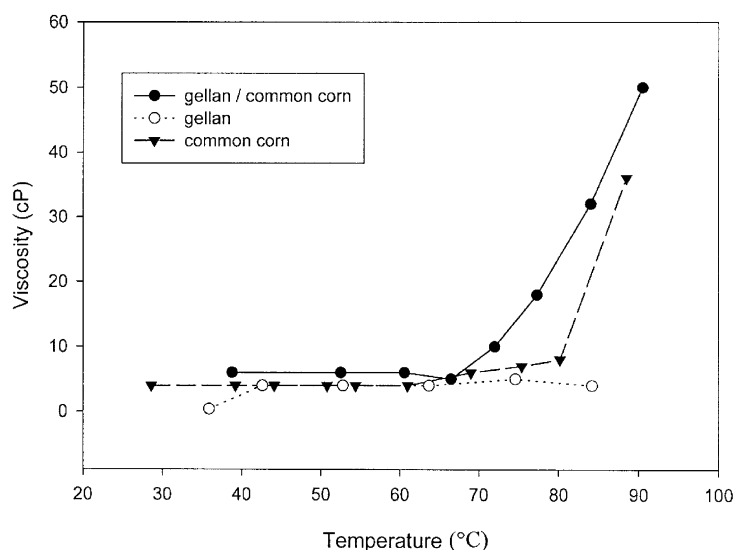


Fig. 2. Brookfield viscosities in centipoises (mPa s) of a suspension of normal maize (common corn) starch granules (3.6%) in water and in a gellan solution (0.4%) and the gellan solution alone heated to 90 °C.

(total weight 28 g, including starch) in an aluminum sample container and stirred manually for 20–30 s before insertion into the RVA.

Phosphorylated common corn starch (ccs) was prepared using sodium tripolyphosphate by following the procedure described by Paschall (1964).

3. Results and discussion

Brookfield viscosities of normal maize starch (ccs) suspended in water, ccs in CMC solution, and CMC solution itself during heating from 35 to 90 °C are plotted in Fig. 1. A slight increase in viscosity (only about 7 mPa s (cP)) for a suspension of normal maize starch in water was detected as the temperature increased from 72 to 82 °C; then a sharper increase in viscosity (40 cP) was detected from 82 to 90 °C. The viscosity of the sodium CMC solution dropped gradually from 80 to 40 cP as it was heated from 35 to 90 °C. The curve for a suspension of normal maize starch in the CMC solution first showed a loss in viscosity similar to that of the CMC solution as it was heated from 35 to 58 °C, then an increase of 60 cP from 58 to 82 °C (first-stage increase), followed by a sharper increase in viscosity (second-stage increase) which was greater than the sum of the separate viscosities produced by the starch and CMC. These results indicated that the Brookfield viscometer was suitable for revealing the synergistic effects in viscosity between a starch and a gum at concentrations that give viscosities that are too low to be detected by the usual instruments used to determine starch pasting curves. They also revealed that starch itself could produce a first-stage viscosity increase that might be undetectable by the usual instruments and that, therefore, the early-stage viscosity increase seen with starch–gum pairs is a matter of magnitude. Thirdly, it

confirmed that synergistic effects in viscosity exist between normal maize starch and CMC.

After obtaining these results, suspensions of normal maize starch, with and without added CMC, were heated on the hot stage of a microscope. No change in gelatinization temperature was observed, indicating that the first-stage viscosity increase was not due to a lowering of the gelatinization temperature. The literature on the effect of an added polysaccharide on gelatinization of granules as examined by DSC supports this finding, although in DSC experiments there must be a larger concentration of starch and, hence, a smaller proportion of water or gum/hydrocolloid solution. Kim and Setser (1992), and Rojas et al. (1999) reported a slight increase in gelatinization onset temperature when wheat starch was heated in solutions of seven different neutral and anionic gums. Yoshimura, Takaya, and Nishinara (1999) reported the same for normal maize starch and xyloglucan. Yoshimura et al. (1999), and Kim and Wang (1999) reported an increase in the peak temperature, while Cameron, Sansom, and Donald (1993), Biliaderis, Arvanitoyannis, Izydorczyk, and Prokopowich (1997), and Rojas et al. (1999) found that gums had little or no effect on the peak gelatinization temperature of wheat and waxy maize starches. No one has reported that gums decrease either the onset or peak temperatures of gelatinization as determined by DSC any significant amount.

In this work, the control starch appears to paste at a higher temperature than normally observed. This is likely due to the much slower, stepwise rate of heating which allows annealing between heating cycles. However, all suspensions were heated in almost identical, and there was a control run with every set; so comparisons are valid.

The same type of experiment was done with gellan and normal (Fig. 2) and waxy (Fig. 3) maize starches. The results for normal maize starch are almost identical to

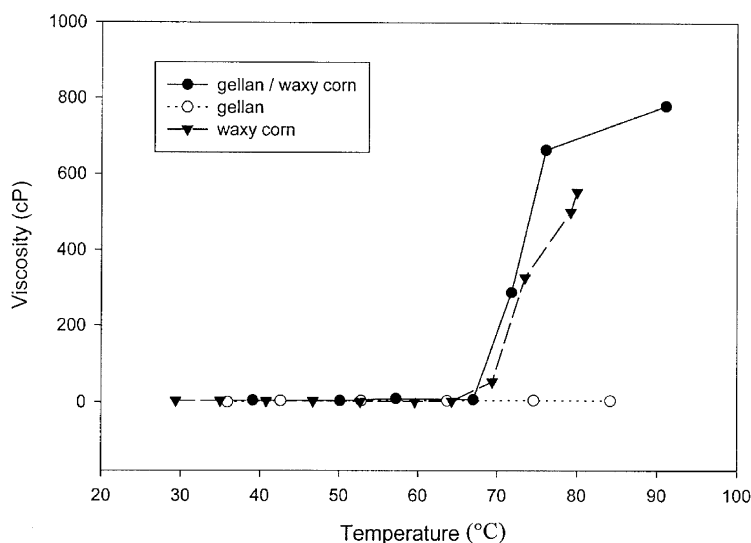


Fig. 3. Brookfield viscosities in centipoises (mPa s) of a suspension of waxy maize (corn) starch granules (3.6%) in water and in a gellan solution (0.4%) and the gellan solution alone.

those observed with CMC (Fig. 1), with the viscosity increase when gellan was present beginning at a temperature about 15 °C lower than the onset temperature of viscosity increase in the absence of the gum (Fig. 2). However, there was only a small lowering of the onset viscosity-increase temperature when a suspension of waxy maize starch was heated in the presence of gellan (Fig. 3). The hypotheses at this point were that the earlier onset of viscosity increase when the gum was present was due to an interaction between gum (CMC, gellan) molecules and leached starch molecules and that amylose leaches from granules early in the pasting process while amylopectin leaching does not occur until just before pasting. Alternatively, gellan could have reduced the pasting temperature of waxy maize starch a degree or two. Additional evidence for the hypotheses was sought.

Similar experiments were conducted using various starches and gums. ‘Yes’ or ‘no’ in Table 1 refers to whether a significant earlier onset of viscosity increase

upon heating was observed for a starch suspension in a gum solution compared to a starch suspension in water. From the results, it is concluded that a significant viscosity increase before pasting existed only with certain starch–gum pairs and that xanthan probably interacted with all starches examined. The viscosity profiles for normal maize starch, waxy maize starch, normal rice starch, and waxy rice starch plus gums are given in Figs. 4–7, respectively. The patterns suggest that interactions between specific gums and specific molecules leached from granules are responsible for the early-stage viscosity increases when starches are cooked in gum solutions and that amylose–gum interactions are probably mainly responsible. Table 1 also suggests that potato and tapioca amyloses behaved differently from cereal amyloses (Figs. 8 and 9). For wheat starch at the low concentration used in this research, an early viscosity increase was not detected with CMC and sodium alginate (Fig. 10), a result different from that

Table 1
Observation of a viscosity increase preceding pasting for starch–gum pairs

Starch	CMC	Xanthan	Guar	Gellan	Alginate	ι -c ^a	κ -c ^b	HPMC
Normal maize	Yes	Yes	Yes	Yes	Yes	No	No	No
Waxy maize	Yes	Yes	Yes	yes	No	No	No	No
Normal rice	Yes	Yes	Yes					
Waxy rice	No	Yes	Yes		No			
Potato	No	Yes			No			No
HPCCS ^c	No	Yes						
Tapioca	No	Yes	No		No			
Mungbean	No	Yes						
Wheat	No	?	Yes		No			

^a ι -Carrageenan.

^b κ -Carrageenan.

^c Hydroxypropylated common corn starch, DS ~ 0.1.

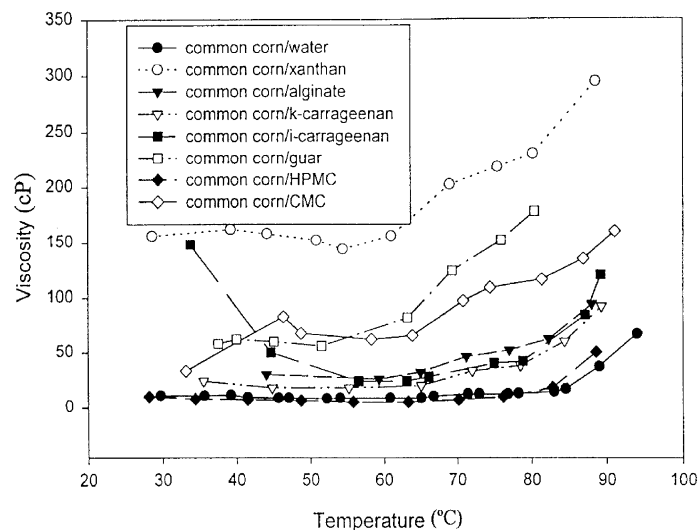


Fig. 4. Brookfield viscosities in centipoises (mPa s) of suspensions of normal maize starch granules (3.6%) in water and in various gum solutions heated from 30–35 to 90–95 °C.

reported from studies in which higher concentrations of starch and gum were used (Crossland & Favor, 1948).

It was decided to try to determine how amylose interacts with gum molecules to produce the first viscosity increase. It is known that, at temperatures below the gelatinization temperature, amylose can and does leach from starch granules (Tester & Morrison, 1990; Young, 1984), but many of these leached amylose molecules are insolubilized by retrogradation. Questions that might be asked are the following. Is it possible that gum molecules interact with amylose molecules partially leached from granules and adhere partially swollen granules (Model A) (Abdulmola et al., 1996)? Do gum molecules interact with leached amylose molecules, produce a viscosity increase via synergism, and/or prevent retrogradation (Model B)?

To determine which model was more feasible, normal

maize starch granules (3.6%) were dispersed in CMC solution (0.4%) and in water, and the suspensions were held at 65 °C for 3 h. Then a drop from each suspension was stained with iodine solution and examined with a microscope. When normal maize starch granules were heated in water, amylose was only observable at the surface or near the periphery of granules, similar to what has been reported (Jacobson, Obanni, & BeMiller, 1997). In contrast, when normal maize starch granules were heated in CMC solution, amylose was found throughout the solution. This indicated that Model B is more likely true. It was also noticed that the granules were equally swelled in both cases, i.e. no size differences could be identified visually. The suspensions were also mixed, then centrifuged, and amylose concentrations in the supernatants measured by the blue value method (Gilbert & Spragg, 1964; Kavitha & BeMiller, 1998). There

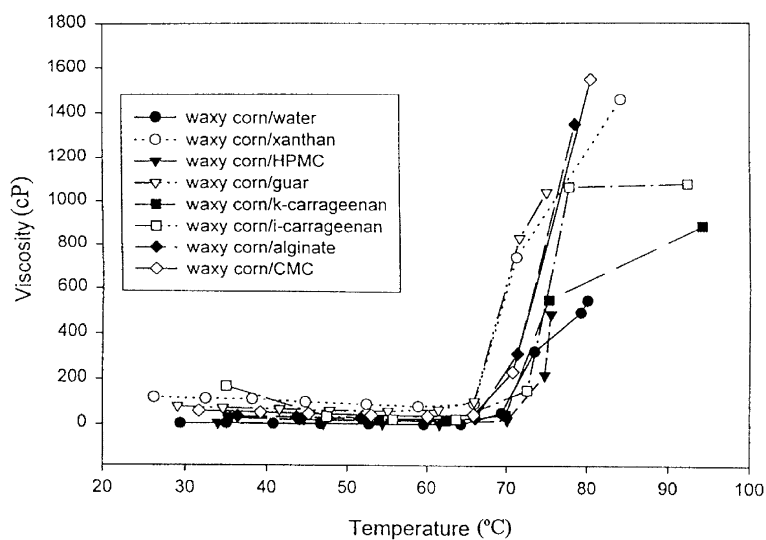


Fig. 5. Data like that in Fig. 2 for waxy maize (corn) starch.

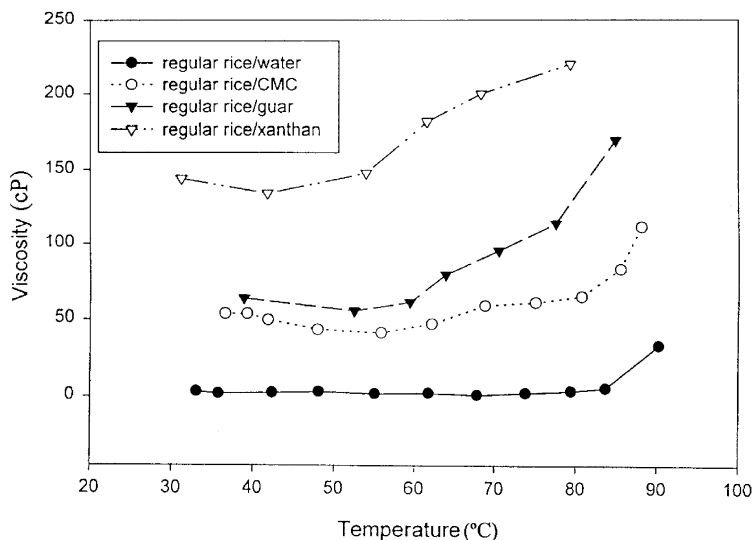


Fig. 6. Data like that in Fig. 2 for normal rice starch.

was no significant difference between the amounts of amylose leached out in water or in CMC solution. Therefore, the main difference between the two systems was the location of amylose. It was concluded that it was likely that interactions between amylose and CMC molecules were at least partially responsible for the first-stage viscosity increase and that the molecular basis was an association between maize amylose and CMC that increased apparent molecular size and viscosity, perhaps also preventing retrogradation and/or interaction with the granule surface.

Similar leaching experiments were done with tapioca, potato, and wheat starches at 57 °C (a temperature below the normal pasting temperature). The results were similar to those obtained with normal maize starch. Tapioca, potato, and wheat amyloses were found throughout the CMC solution, just as maize amylose was, but tapioca, potato, and

wheat starches did not give an early viscosity increase when granule suspensions were heated in CMC solutions, suggesting that tapioca, potato, and wheat amylose did not interact as strongly with CMC as did maize amylose.

Table 2 lists the extents of viscosity changes during pasting (the second stage viscosity increase for 'yes' pairs and the only viscosity increase for the 'no' pairs of Table 1), a result mainly from synergistic effects in the interactions between amylopectin and gums. Of the gums examined, only xanthan and guar gum interacted with all starches tested (except potato starch) and produced a significant viscosity increase. Of starches examined, only waxy maize, waxy rice, and tapioca starches showed a significant viscosity increase with most gums tested (except HPMC).

Although literature on the subject is sparse, it has been recognized for more than a decade that amylopectin can also

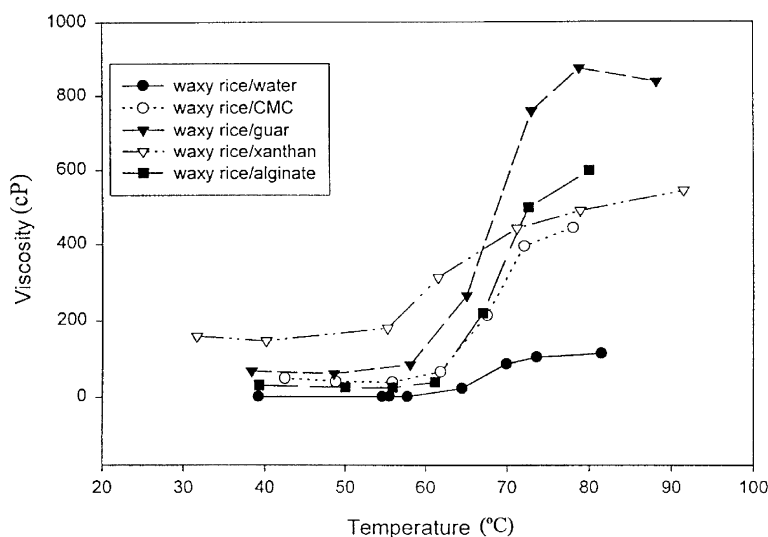


Fig. 7. Data like that in Fig. 2 for waxy rice starch.

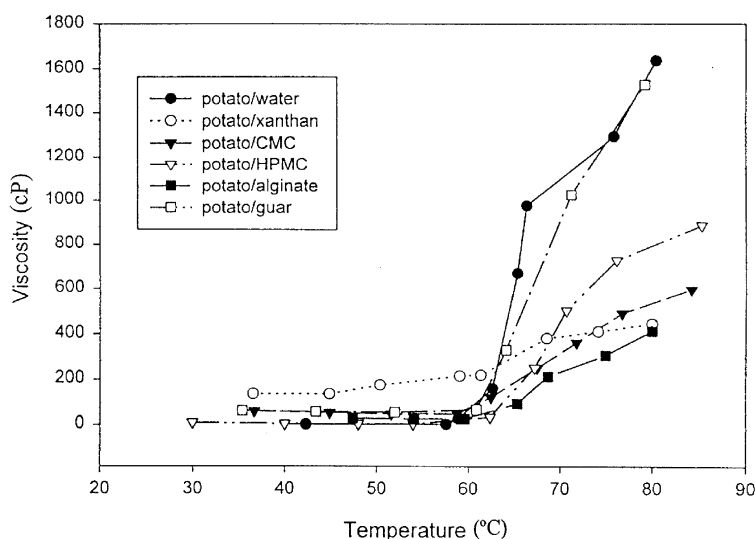


Fig. 8. Data like that in Fig. 2 for potato starch.

leach from wheat, barley, and maize starch granules held in suspension at temperatures below the gelatinization temperature (Tester & Morrison, 1990). Recently, we have found that considerable modified amylopectin leaches from hydroxypropylated normal maize starch ($MS \approx 0.1$), even though the amylose is more highly substituted (Shi & BeMiller, in press).

Fig. 8 (and Table 2) illustrates that suspensions of potato starch in anionic gum solutions (CMC, sodium alginate, xanthan, carrageenan) had a much lower viscosity during pasting (at 80–85 °C) than did a potato starch in water suspension, while a guar gum (neutral gum) solution had little effect. Microscopic examination revealed that potato starch granules were broken after being stirred slightly in water at 65 °C for 3 h, while potato starch granules suspended in solutions of anionic gums and subjected to

heating under the same condition were mostly unbroken, i.e. intact. (See further for heating potato starch granules to 95–100 °C in solutions of anionic gums.) Two possible reasons for the decreased viscosity were proposed: (a) negatively charged gum molecules prevent the swelling and breaking of potato starch granules; (b) negatively charged gum molecules somehow reduced the viscosity of the pasted starch system.

To determine whether negative charges affected potato starch pasting viscosity, potato starch was cooked in salt solutions (Fig. 11). Peak viscosities were greatly reduced when NaCl was present in solution (while dilute salt solution had no effect on common corn starch). To determine whether it was the anions or the cations that caused the viscosity changes, CsCl and NaI were also used separately. (Molar concentrations of the salts were kept constant.) Peak

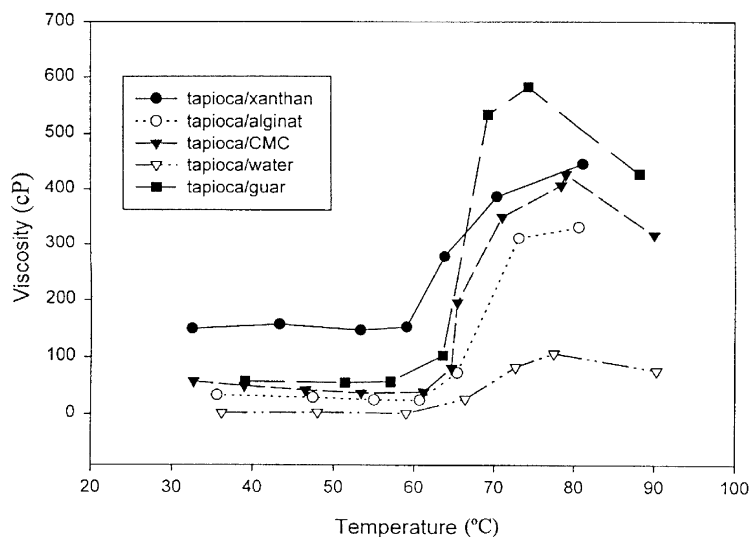


Fig. 9. Data like that in Fig. 2 for tapioca starch.

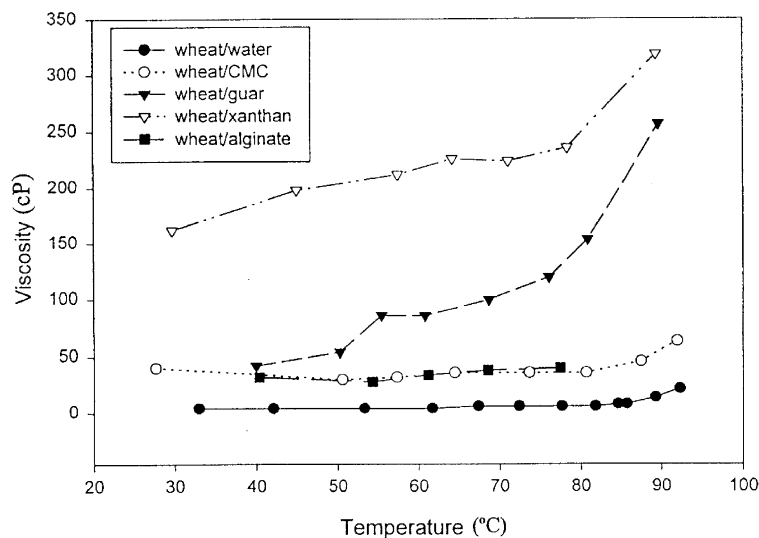


Fig. 10. Data like that in Fig. 2 for wheat starch.

viscosity did not change when Na^+ ions were replaced with Cs^+ ions, while peak viscosity did change when Cl^- ions were replaced with I^- ions, indicating that it was the anions that resulted in peak viscosity reduction.

Normal maize starch was phosphorylated using sodium tripolyphosphate ($\text{DS} < 0.01$) to give it, like potato starch, negative charges without crosslinking. Xanthan (0.1%) increased the peak viscosity of normal maize starch slightly (Fig. 12), but greatly reduced the peak viscosity of normal maize starch phosphate (common corn starch phosphate, p-ccs). Another interesting observation with p-ccs–gum interaction is shown in Fig. 13. When p-ccs was heated in an ι - or λ -carrageenan solution, reduced peak viscosity was observed, just as with xanthan, and a reproducible second viscosity peak was observed when the temperature was held at 95 °C. The second peak was not observed with κ -carrageenan solution.

RVA peak viscosities of potato starch cooked in gum solutions are given in Table 3. The RVA peak viscosity of

potato starch in water was 11,200 cP. Guar gum (at concentrations of 0.4 and 0.1%) effected little change, while peak viscosity was greatly reduced by sodium alginate, CMC, pectin, xanthan, κ -carrageenan, λ -carrageenan, and ι -carrageenan, all anionic, i.e. negatively charged, gums. When the concentration of xanthan was increased from 0.1 to 0.4%, and the same starch concentration was kept (7.5%), viscosity was further decreased, i.e. there was a definite concentration dependence; but increasing the concentration of carrageenan from 0.1 to 0.4%, increased the peak viscosity, probably simply from an increased viscosity due to increased gum concentration.

To investigate further the effect of negatively charged gum molecules on potato starch granules during cooking, potato starch granules were dusted into 10 ml of 0.4% sodium CMC, sodium alginate, and xanthan solutions and water (control) in screw-capped tubes and heated in a boiling water bath for 10 min; then the suspensions were dyed with iodine solution and examined with a light microscope

Table 2

Extents of viscosity change during pasting (a, increase greatly; b, increase moderately or slightly; c, no change; d, decrease slightly; e, decrease greatly compared to when gum was absent)

Starch	CMC	Xanthan	Guar	Gellan	Alginate	ι -c ^a	κ -c ^b	HPMC
Normal maize	a	a	a	b	b	b	b	c
Waxy maize	a	a	a	b	a	a	b	c
Normal rice	b	a	a					
Waxy rice	a	a	a		a			
Potato	e	e	c		e			d
HPCCS ^c	b	a						
Tapioca	a	a	a		a			
Mungbean	b	a						
Wheat	b	b	a		b			

^a ι -Carrageenan.

^b κ -Carrageenan.

^c Hydroxypropylated common corn starch, $\text{DS} \sim 0.1$.

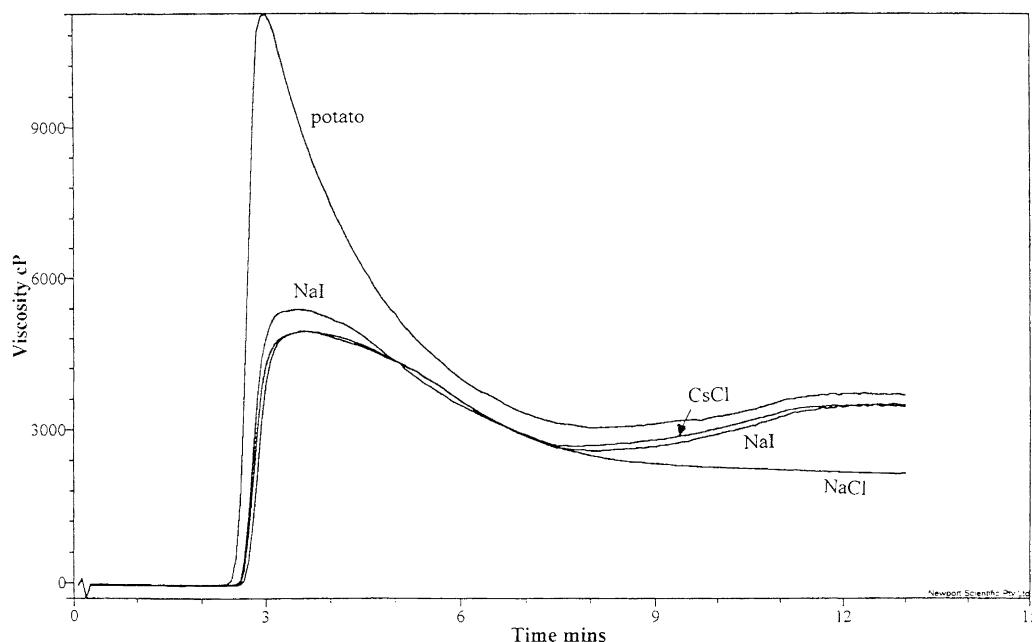


Fig. 11. RVA curves of potato starch in 0.017 M solutions of NaCl, NaI, and CsCl.

(Obanni & BeMiller, 1995). Potato starch granules mostly disintegrated when heated in water to 95–100 °C with minimal shear. In an anionic gum solution (Fig. 14), the granules were more intact, although there was leaching of polymer molecules. The latter granules had no birefringence, indicating that they were gelatinized, but the pasting process appeared to have been arrested before completion. Interestingly, many granules had an opening at one end, indicating that that is where the granule envelope first

breaks. Some granules, primarily the smaller ones, had a blue core and a red-brown shell, indicating that amylose had leached from the outside layer, but there was a definite depth to this layer, i.e. no indication of diffusion of amylose molecules from the inner area to the zone of leaching. Differences in behavior of large and small granules are another indication of granule heterogeneity. Sekine (1996) also reported that starch granules heated in a xanthan solution were not disintegrated and that the resulting mixture

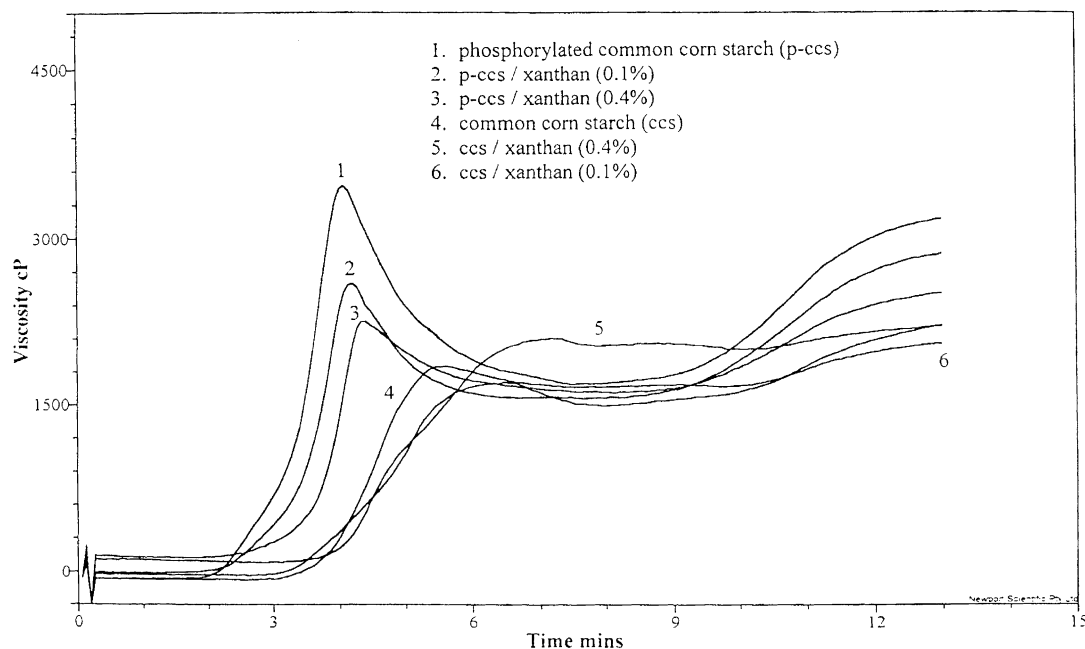


Fig. 12. RVA curves of normal maize (common corn) starch and phosphorylated normal maize (common corn) starch in xanthan solutions (starch/gum percentages 3.6:0.4 and 3.9:0.1).

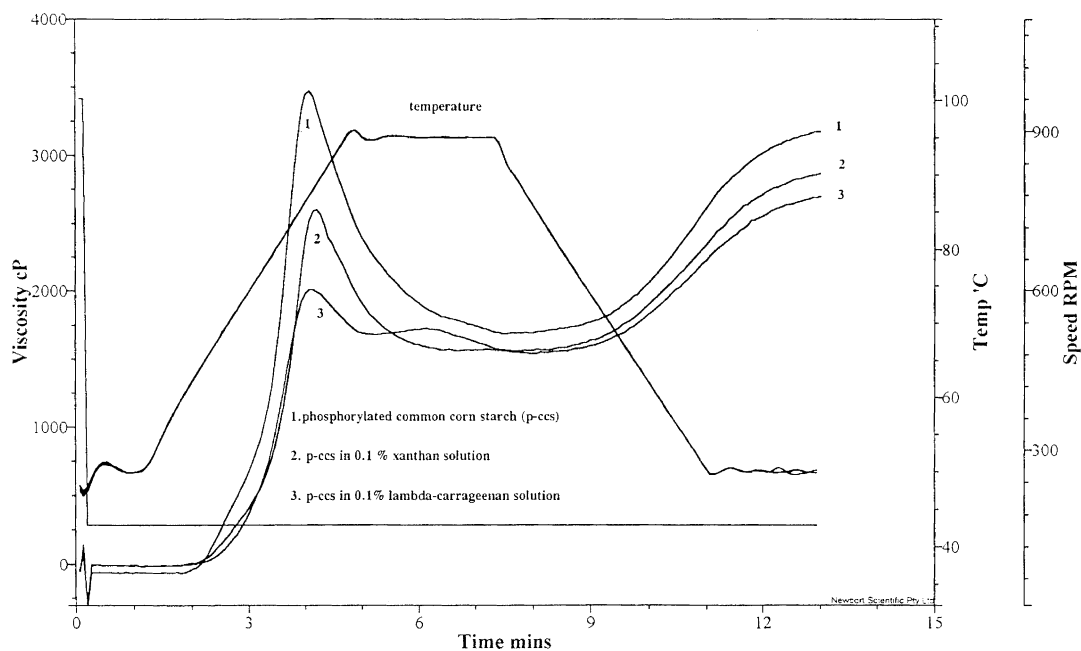


Fig. 13. RVA curves of phosphorylated normal maize (common corn) starch (3.9%) in xanthan and λ -carrageenan (0.1%) solutions.

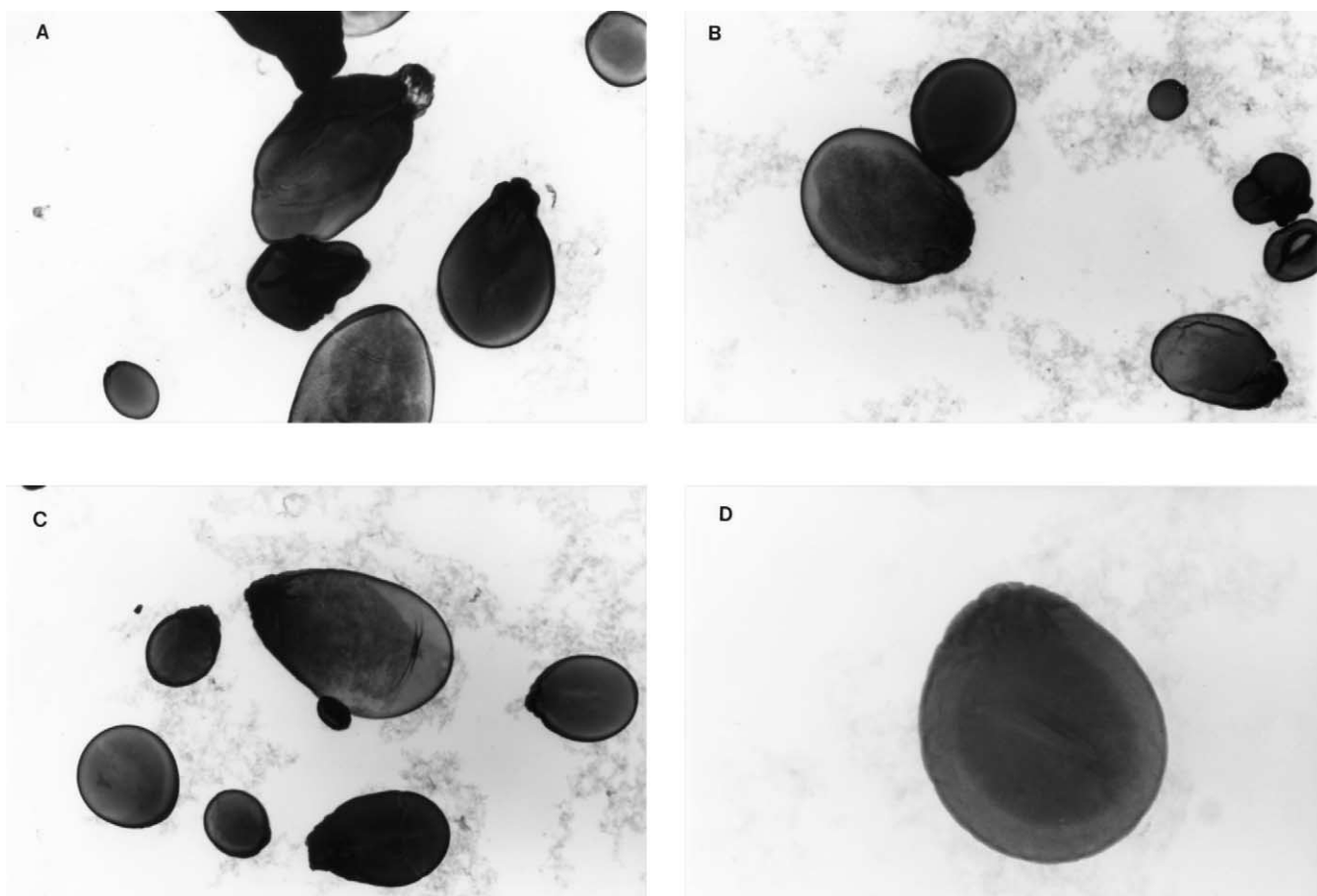


Fig. 14. Photographs of potato starch granules stained with iodine after a suspension in an anionic gum solution (0.4%) was heated in a boiling water bath for 10 min. (A) granule after heating in an alginate solution showing granule matrix material protruding from an apparent rupture in the outer envelope; (B) and (C), granules after heating in an alginate solution showing less protrusion of contents and smaller granules exhibiting a blue core surrounded by a reddish area devoid of blue-staining material; (D) an enlargement of a granule after heating in a CMC solution from which granule amylose has leached from the outer layer and from which contents are just beginning to exude from the weak area.

Table 3
RVA peak viscosity of potato starch (7.5%) in gum solutions

Gum	Gum concentration (%)	Peak viscosity (cP)
Water	0	11,200
Guar	0.1	10,900
Guar	0.4	11,100
Alginate	0.4	5240
CMC	0.4	5010
Pectin	0.4	3780
Xanthan	0.1	4230
Xanthan	0.4	2810
κ-Carrageenan	0.1	3540
κ-Carrageenan	0.4	3865
λ-Carrageenan	0.1	3340
λ-Carrageenan	0.4	4030
ι-Carrageenan	0.1	3260
ι-Carrageenan	0.4	3820

had properties of a suspension of swollen granules in a xanthan solution continuous phase.

Because of these results, obtained at low concentrations, we favor intermolecular associations between leached molecules (primarily amylose molecules) and gum molecules as the explanation for the viscosity increase that occurs in dilute starch–gum systems before the onset of pasting. Retardation of granule pasting/destruction and leaching of amylose seems to be the cause of the delay in pasting and reduction in peak viscosity when negatively charged starches are heated in solutions of anionic gums. Detailed studies using this technique coupled with dynamic rheological and other analyses are needed to determine the exact molecular basis or bases for these phenomena.

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