Paste and Gel Properties of Prime Corn and Wheat Starches With and Without Native Lipids[1]

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ABSTRACT

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Corn starch showed higher fresh paste consistency than wheat starch, and, after aging 24 hr, its gels were more firm at 6% solids or less. Between 7 and 30% solids, gels of wheat starch were more firm than those of corn starch. The lipids in wheat and corn starches were reduced 88–97% by boiling three times in four parts of a 3:1 (v/v) mixture of ethanol/water. Iodine-binding capacity of corn and wheat starches was highest after extraction with 75% *n*-propanol at 100° C compared to other solvents. Soxhlet extraction with methanol removed 93% lipid from corn starch, but only 78% from wheat starch as judged by iodine-binding capacity; hot 75% ethanol removed 97% lipid from wheat starch, but only 88% from corn starch. Amylograms of the low-lipid starches in 1% carboxymethylcellulose showed an initial, rapid increase in consistency as temperature rose 10° C beyond gelatinization. Then, consistency increased slowly and steadily up to 95° C. This initial rapid increase coincided with the rapid release of amylose into the soluble phase of the paste. Amylograms of the low-lipid starches in water showed a decrease in pasting temperature, no pasting peak, and reduced consistency and setback. When prime wheat starch was impregnated with 2% wheat starch lipids, the pasting peak and consistency in the amylograph increased, and a strong second peak was observed during the cooling cycle. When the same amount of wheat starch lipid was added to the starch paste after it reached 95° C in the amylograph, the peak during the cooling cycle was markedly reduced, as was setback. Setback was not observed at 2% added lipids. Wheat lipids decreased gel strength and syneresis somewhat during storage at 4° C, but they did not affect syneresis during freeze-thaw treatment.

Corn, potato, wheat, tapioca, and waxy maize are the five commercially important starches around the world (Swinkels 1985). Normal corn and wheat starches resemble each other in properties and differ markedly from the other three. The selection of corn or wheat starch depends on differences in cost, granule size, color, minor constituents, and pasting and gelling properties. On the molecular level, corn and wheat amyloses differ in structure (Takeda et al 1987). Furthermore, their amylopectins probably have different unit-chain distributions (Hizukuri 1986), a conclusion indicated by comparing distributions reported in other investigations on corn (Praznik et al 1987, Takeda and Hizukuri 1988) and wheat (Kobayashi et al 1986).

Monoacyl lipids are known to play a major role in the paste and gel behavior of wheat and corn starches (Eliasson 1986, Dengate 1984, Morrison and Milligan 1982). Furthermore, starch lipids may be a nuisance when converting starch to sweeteners (Bowler et al 1985). In the past, several investigators examined wheat and corn starches that were "defatted" by hot aqueous methanol or *n*-butanol (Melvin 1979, Lorenz 1976, Goering et al 1975, Medcalf et al 1968). For food use, ethanol may be more suitable than other aliphatic alcohols to remove starch lipids (Morrison and Coventry 1985), since it is readily produced by starch manufacturers and is less toxic.

Our objective was to compare the paste and gel properties of prime corn and wheat starches, both before and after extraction of lipid with hot aqueous ethanol.

MATERIALS AND METHODS

Starches

Prime wheat starch and drum-cooked-dried wheat starch were obtained from Midwest Grain Products, Atchison, KS; dent corn starch and drum-cooked-dried corn starch were obtained from A. E. Staley Manufacturing Co., Inc., Decatur, IL. The wheat and corn starch samples contained 0.09% and 0.07% Kjeldahl nitrogen, respectively. Hydroxypropyl wheat starch was provided by C. C. Maningat.

Granular cold-water-soluble corn and wheat starches were supplied by A. E. Staley. Extrusion-cooked corn and wheat starches were produced in a Brabender laboratory-scale extruder (3/4-in. diameter, length/diameter = 25:1) using a screw with a compression ratio of 4. The starches were tempered to 18% moisture and cooked at 100 rpm, with zone one set at 25° C, zone two at 100° C, and zone three at 175° C.

Extraction of Lipids from Starch

Total lipids in starch were estimated gravimetrically after extraction with a 3:1 (v/v) mixture of *n*-propanol water at 100° C (Morrison and Coventry 1985). Starch (2.0 g) and *n*-propanol/water (25 ml) were placed into each of five screw-cap, glass centrifuge tubes (25 × 150 mm). Thin polyfilm was wrapped around the threads of the tubes, the caps were screwed on tightly, and the tubes were heated in a boiling water bath for 2 hr. The tubes were cooled and the contents filtered through a sintered glass funnel. The starch was extracted twice more with hot *n*-propanol/water, and the filtrates combined and evaporated to dryness under vacuum at approximately 50° C. The residue was extracted (3 × 10 ml) with a mixture of chloroform/methanol (2:1, v/v), and the combined extracts were filtered and evaporated to dryness. The lipids were dried by three evaporations from absolute ethanol before weighing. Lipid contents were reported on a dry starch basis.

Large quantities of low-lipid starch were prepared by heating starch (100 g) in a boiling (81° C) mixture (3:1, v/v) of ethanol/water (400 ml) for 6 hr. The mixture was centrifuged, and the sedimented starch was extracted twice more. The combined supernatant layers were filtered through Whatman No. 4, and the filtrate was evaporated to dryness under vacuum. The residue was extracted 3× with 50 ml of a mixture of chloroform/methanol (2:1, v/v), and the extracts were filtered through Whatman No. 4 filter paper. The filtrate was evaporated under vacuum, and the residual lipids were dried by three additions and evaporations of ethanol.

Amylose Determination

Amylose was isolated from wheat and corn starch using the method of Adkins and Greenwood (1969). The amylose samples were assumed to be pure after three crystallizations of the *n*-butanol complex as reported by Takeda et al (1984). Yields were 10–15% based on starch. Amylose in starch and in the solubles leached from starch by hot water was determined by iodine-binding capacity (Schoch 1964b).

Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) was done with a Perkin

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Elmer DSC-2 equipped with a FTS System Flexicooler with temperature controller. Indium was used to calibrate the instrument. Samples of starch (2–3 mg) and water (1:3, w/w) were sealed in aluminum pans and heated at 10° C/min from 2 to 120° C at a sensitivity of 0.5 mcal/sec.

Consistency of Starch Pastes

Amylograms were recorded on a Brabender Viscograph-E (C. W. Brabender Instruments, Inc., Hackensack, NJ) according to the general procedure described by Tipples (1980). The initial and final temperatures in the amylograms were 30° C, and pastes were held at 95° C for 10 min between the heating and cooling cycles. Some curves were obtained using 1% carboxymethylcellulose (CMC) to prethicken the aqueous phase (Crossland and Favor 1948). The amylogram of 1% CMC alone was subtracted from an amylogram of starch in 1% CMC.

Amylograms of wheat starch also were recorded in the presence of added wheat starch lipids. Wheat starch was either impregnated with the lipids prior to recording the amylograms, or the lipids were added to the amylograph bowl when 95° C was reached on the heating cycle. To impregnate the starch with lipids, a solution of wheat starch lipids (0.5, 1.0, and 2.0 g) in absolute ethanol (127 ml) was diluted with water (42 ml) and kept at 40° C; then wheat starch (56 g with 11% moisture) was added, and the mixture was stirred for 6 hr. The solvents were removed by evaporation under vacuum, and water in the starch was removed by azeotropic evaporation (2×) using absolute ethanol. The starch was spread in a thin layer and held overnight under ambient conditions (25° C and approximately 70% relative humidity). When lipids were added to a wheat starch paste at 95° C, or, in one instance, directly to the amylograph bowl at the start (25° C) of the run, a 1.0-, 2.0-, or 4.0-ml aliquot of a solution of wheat lipids (3.79 g) in ethanol (20 ml) was added to a total of 450 g of paste containing 7.5% starch solids.

The viscosity of a starch (5 g, dry solids) solution in a 9:1 (v/v) mixture (500 ml) of dimethyl sulfoxide and water was determined with a Brookfield viscometer using a Helipath stand with spindle A at 12 rpm.

Swelling Power and Solubility of Starch and Iodine-Binding Capacity of Solubles

Swelling power and solubility were determined using a modification (Kainuma et al 1967) of the method of Leach et al (1959). Mixtures of starch (1 g) and water (50 ml) were heated in plastic centrifuge tubes at 65, 75, 85, and 95° C for 40 min. During heating, the tubes were shaken gently to prevent clumping of the starch. Immediately after centrifuging (3,000 rpm), the carbohydrate in the supernatant was determined colorimetrically (Dubois et al 1956), and the weight of the sediment was recorded. Swelling power was the ratio of the wet mass of the sedimented gel to the dry matter in the gel, and solubility was the percentage of starch dissolved in the continuous fluid phase. The amylose in the soluble phase was determined by iodine-binding capacity, while λ_{max} of the iodine complex was determined according to Chrastil (1987).

Gel Texture

All gel texture measurements were done on quadruplicate samples. Starch gels with 5–10% dry solids were prepared by cooking in the amylograph in water or in 1% CMC at 1.5° C/min from 30 to 95° C and then holding at 95° C for 5 min. Hot pastes were poured into shallow petri dishes (50 × 17 mm) or deep dishes (50 × 30 mm). The depth of each dish was increased approximately 5 mm by taping aluminum foil around its rim. The petri dishes were covered, and the gels were stored for 24 hr at 4 and 25° C. After the aluminum foil was removed, a smooth, freshly cut surface was obtained by removing the excess gel above the rim with a wire cheese cutter.

The texture of gels (5–10% starch solids) was determined using a Voland-Stevens texture analyzer (Voland Corp., Hawthorne, NY) fitted with a chart recorder. The thin gels (17 mm) were compressed at a speed of 0.2 mm/sec to a distance of 4 mm using a cylindrical plunger (diam. 25.4 mm) with the chart recorder speed at 20 cm/min. The peak height at 4 mm compression was called firmness. The deep gels (approximately 30 mm) were used to obtain load-penetration curves (Marrs et al 1980). A spherical probe (diam. 12.7 mm) was advanced at a rate of 0.5 mm/sec to a distance of 15 mm into a gel, and then retracted at the same speed. The firmness of a gel was measured by the load at a depression distance of 2.5 mm. The load at the breaking point of the gel was reported, and stickiness, which was indicated by the negative load portion of the curve, was noted.

Starch gels containing 10–30% starch solids were prepared according to Maningat (1986), who modified the method of Krusi and Neukom (1984). Starch was blended 1 min at low speed with 1% aqueous CMC, and the suspension (total approximately 150 ml) was degassed in a vacuum desiccator for 10 min. Twenty-four grams of the thick slurry of suspended starch granules was added to each of five small petri dishes. After being covered, the petri dishes were heated at 100° C for 110 min. Upon removal from the oven, the lids on the dishes were replaced with ones lined with paper. After being cooled for 2 hr at 25° C, the lids were switched to nonlined ones, and the gels were stored 24 hr at 25° C. Firmness of the 10–30% gels was measured using an Instron universal testing machine at a crosshead speed of 25 cm/min and a chart speed of 50 cm/min. Firmness was the load at a depression of 2 mm, and breaking point was the load when the gel fractured.

Gel Stability Under Cold Storage and Freeze-Thaw Conditions

After the firmness measurement was completed on a thin gel, the gel was subjected to cold storage and freeze-thaw conditions. For cold storage, the replicate samples of a gel were transferred to small trays that had been tared. The gels were cut into four pieces, and the loaded trays were wrapped tightly with Parafilm and placed in plastic bags that were sealed and stored at 4° C. After three, seven, and 10 days, duplicate gel samples were removed. The gel solids were separated from the water of syneresis using a spatula, and syneresis was calculated as the percent water released based on initial gel weight.

To measure freeze-thaw stability, gels were frozen at −20° C for 24 hr and thawed for 4 hr at 25° C. After one to three freeze-thaw cycles, water of syneresis from the gel was collected by vacuum filtration on a sintered (M) glass funnel fitted with Whatman No. 4 filter paper.

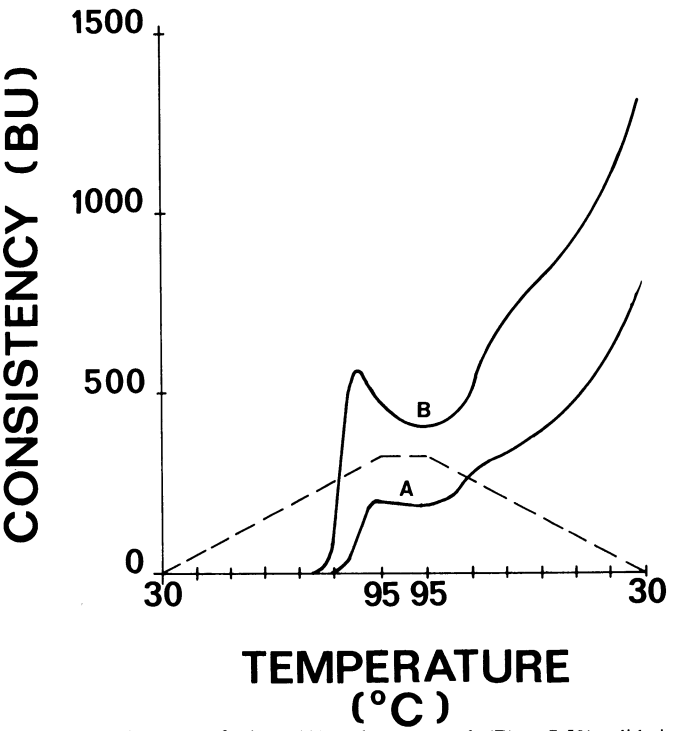

Fig. 1. Amylograms of wheat (**A**) and corn starch (**B**) at 7.5% solids in water. The dashed line is the temperature profile. BU = Brabender units.

RESULTS AND DISCUSSION

Pasting of Commercial Starches

Fresh pastes from corn starch are known to have a higher consistency than those from wheat starch (Doublier et al 1987, Swinkels 1985, Mazurs et al 1957), the commercial samples of prime starch used in this work conformed to that norm (Fig. 1). Addition of 0.04% mercuric chloride, an α-amylase inhibitor, did not change the amylograms. The higher consistency of corn starch prevailed in several forms of precooked starch (Fig. 2), but the difference in paste consistency decreased as starch granules were destroyed in the precooking process. Thus, granular cold-water-soluble (GCWS) starches showed major differences in cold-paste consistency, whereas extruded wheat and corn starches had much smaller differences. The GCWS starches were comprised of mostly intact granules (Jane et al 1986), whereas the extrusion-cooked starches contained few granules because of the high shear in the laboratory extruder (Faubion et al 1982).

When the corn and wheat starches were dissolved in a 9:1 (v/v) mixture of dimethyl sulfoxide and water, the viscosity of the corn starch solution was slightly below that of wheat starch. Values for viscosity (determined with a Brookfield viscometer using a Helipath stand with spindle A at 12 rpm) were 31 cps for corn starch, 36 cps for wheat starch, 39 cps for low-lipid corn starch, and 32 cps for low-lipid wheat starch. In dilute solution, the hydrodynamic volume occupied by the amylose and amylopectin molecules controls viscosity. Both starches contained approximately 30% amylose as determined by IBC. The weight-average molecular weight (M_w) of corn amylopectin has been reported to be $40–80 \times 10^6$ daltons (Da) (Erlander and French 1956) and of wheat amylopectin, 400×10^6 Da (Banks et al 1972). Corn amylose gave M_w 120,000 Da (Eberman and Praznik 1975) or number-average molecular weight (M_n) 156,000 (Takeda et al 1987) and wheat amylose M_w 340,000–2,650,000 Da (Young 1984, Banks and Greenwood 1967) or M_n 109,000 (Takeda et al 1987). Swinkels (1985) reported that amylopectin and amylose from both starches have molecular weights of 400×10^6 Da and 0.13×10^6 Da, respectively.

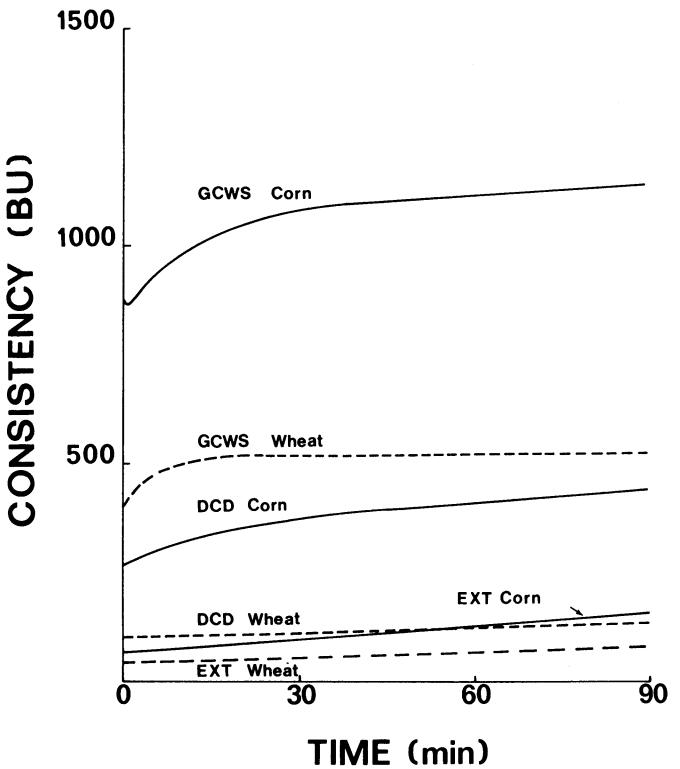

Fig. 2. Cold-paste (30° C) amylograms of "precooked" starches at 7.5% solids. GCWS = granular cold-water soluble; DCD = drum cooked and dried; EXT = extruded at 175° C and 18% moisture. BU = Brabender units.

Swelling power measurements (Table I) confirmed the recent data of Doublier et al (1987) showing that corn starch pastes contain more swollen gel phase than wheat starch pastes at and above, but not below, 75° C. Furthermore, we found 18–21% solubles at 95° C in the continuous phase of the two pastes.

The higher consistency of a freshly made corn starch paste versus wheat starch paste is due to the higher volume of its swollen gel phase, which simultaneously gives a higher concentration of polymer in the continuous phase of the paste (Ghiasi et al 1982). That hypothesis discounts differences in the rigidity of the swollen granules and in the adhesion between amylose in the dispersed phase and the swollen granules (Eliasson and Bohlin 1982).

What accounts for the higher swelling power of corn as opposed to wheat starch? There is some difference (<3%) in the density of corn (1.637 g/cm^3) versus wheat starch (1.595 g/cm^3) (French 1984). Bowler et al (1980) pointed out that during pasting, large wheat starch granules swell little in the direction of the disk's minor axis. On the other hand, spherical corn starch granules appear to swell uniformly (Williams and Bowler 1982). Ultimately, differences in the molecular structures of corn and wheat amylose (Takeda et al 1987), and probably corn (Praznik et al 1987, Takeda and Hizukuri 1988) and wheat (Kobayashi et al 1986) amylopectin are translated into differences in swelling power.

Low-Lipid Starches

Three successive, 6-hr extractions of starch with four parts of boiling 75% ethanol removed 0.52, 0.22, and 0.26 g of lipids from 100 g (dry solids) of wheat starch and 0.55, 0.19, and 0.08 g from corn starch. The lipids extracted represented approximately 90% of the total lipid in the prime wheat starch (1.1% of dry solids) and corn starch (0.91% of dry solids), which was determined by exhaustive extraction with 75% propanol at 100° C (Morrison and Coventry 1985).

Morrison and Milligan (1982) reported that maize starch contains 0.59–0.76% lipids, and wheat starch contains 0.77–1.17% (dry solids basis). The boiling point of 75% ethanol at atmospheric

TABLE I
Swelling Power, Solubility, and Iodine-Binding Capacity (IBC) of the Solubles in 2% Wheat and Corn Starch Pastes

Temperature (° C)	Swelling Power (g/g)		Solubility (%)		Solubles Released from Low-Lipid Starches	
	Prime	Low-Lipid	Prime	Low-Lipid	λ_{max} of Iodine Complex (nm)	IBC[a] (mg/100 mg)
Wheat starch						
65	5.4	5.2	3.5	11.7	625–645	12.2
75	6.5	6.1	8.7	16.6	628–642	14.8
85	6.2	6.9	11.9	18.2	628–645	15.2
95	9.3	7.3	20.0	20.6	620–640	15.5
Corn starch						
65	4.5	3.2	2.9	2.0	615–635	10.1
75	7.1	7.4	7.3	13.3	620–635	12.8
85	8.3	8.2	11.8	15.8	620–635	14.3
95	10.5	9.3	18.1	19.5	620–635	14.1

[a]IBC of wheat amylose was 19.3 mg/100 mg.

pressure is 81° C, which is below the melting point (~105° C) of the lysolecithin-amylose complex of wheat and corn starches at 70–90% moisture content (Biliaderis et al 1985, Kugimiya and Donovan 1981) and below the melting point (> 95° C) of the amylose-myristic acid or palmitic acid complex (Biliaderis et al 1985, Kugimiya et al 1980). Over 90% of the lipid in wheat starch is lysolecithin (Meredith and Dengate 1978), whereas the lipid in corn starch is composed of approximately 60% free fatty acids and 25% lysolecithin (Tan and Morrison 1979). The predominant fatty acids (free and esterified) in wheat starch are palmitic and linoleic, and in corn starch the same two acids predominate with some oleic acid. Ethanol appears to displace the lysolecithin and free fatty acids from the amylose complex when used repeatedly in large excess.

We found that the amount of lipid removed from corn starch depended on pH. When we adjusted the acidity of the corn starch slurry in 75% ethanol from pH 4.3 (commercial sample) to 6.0 using sodium bicarbonate, the yield of lipid decreased from 0.82 to 0.44 g per 100 g of dry starch. The sodium salts of the free fatty acids were apparently difficult to dissolve in hot 75% ethanol.

Differential scanning calorimetry confirmed that boiling 75% ethanol removed most of the lipid in corn and wheat starches without any significant change in their crystalline phases. The low-lipid wheat and corn starches failed to show the amylose-lipid endotherms (Kugimiya and Donovan 1981, Biliaderis et al 1985) at 370° K (97° C) and 364° K (91° C), respectively, which were observed in the thermograms of the prime wheat and corn starches (Figs. 3 and 4). The crystallites in the starches gave the following melting ranges and enthalpies: prime wheat starch, T_m 48–73° C, ΔH + 2.5 cal/g; low-lipid wheat starch, T_m 48–74° C, ΔH + 2.4 cal/g; prime corn, T_m 58–79° C, ΔH + 3.0 cal/g; and low-lipid corn starch, T_m 58–79° C, ΔH + 3.4 cal/g.

The iodine-binding capacity (IBC) of starch is also used to indicate the efficiency of lipid removal by various solvents (Table II). The IBCs of prime-wheat and corn starches were highest (5.8 mg/100 mg) after extraction with hot 75% *n*-propanol. The native lipids in wheat starch reduced its IBC about 20%, which agrees with the estimate that 25% of wheat amylose forms a complex upon heating in water (Wren and Merryfield 1970). In corn starch, the native corn lipids reduced the IBC of KOH-gelatinized corn starch by 40%, which is near the value of 35% reported by Doublier et al

TABLE II
Method of Extracting Starch Lipids and Iodine-Binding Capacity (IBC)[a]

Solvent	Temperature (° C)	Extraction Method	IBC[b] (mg/100 mg) Corn	Wheat
None[c]	...	...	3.5	4.3
None[d]	...	...	4.2	4.5
75% Propanol	100	3 × 2 hr (sealed tube)	5.8	5.8
Methanol	30	24 hr (Soxhlet)	5.4	4.5
95% Ethanol	30	24 hr (Soxhlet)	4.4	4.7
75% Ethanol	81	3 × 6 hr Reflux	5.1	5.6

[a] Dry-solids basis.
[b] IBC of amylose isolated from corn and wheat starches was 19.3 and 19.5 mg/100 mg, respectively.
[c] Gelatinized in 1 *M* aqueous potassium hydroxide unless otherwise noted.
[d] Gelatinized in pressure cooker (110° C for 4 hr).

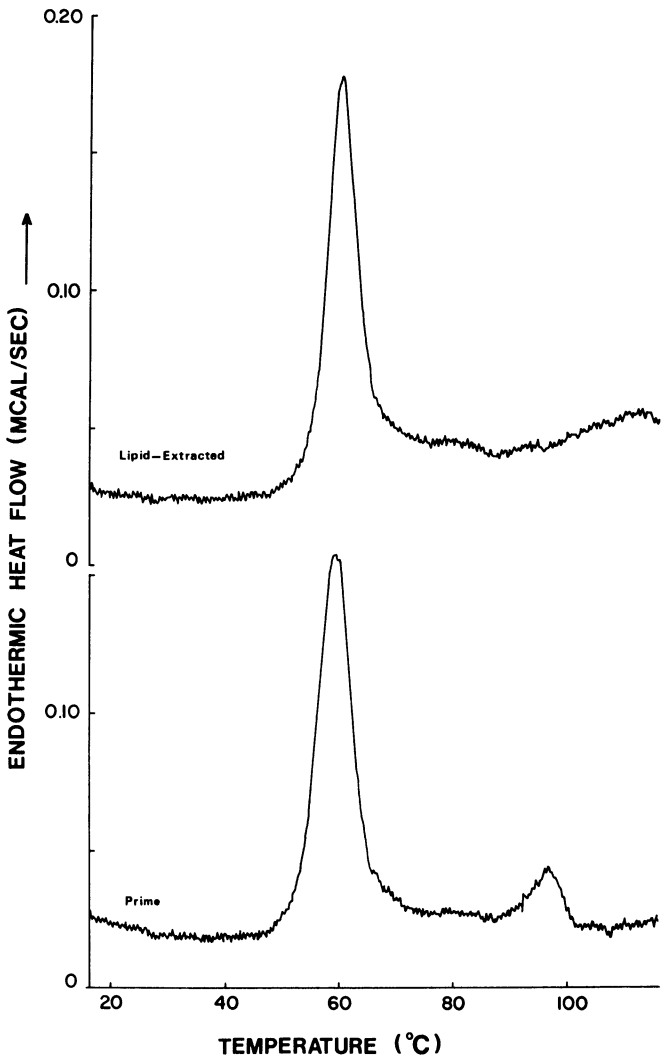

Fig. 3. Differential scanning calorimetry of prime wheat starch and low-lipid wheat starch at a water/starch ratio of 3:1 (w/w).

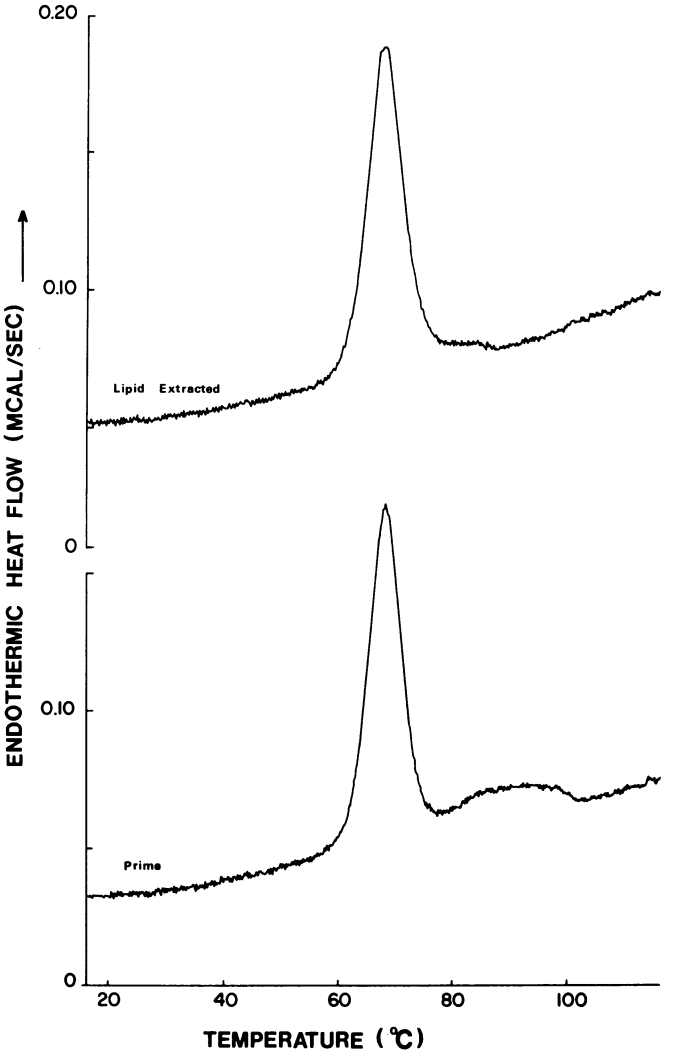

Fig. 4. Differential scanning calorimetry of prime corn starch and low-lipid corn starch at a water/starch ratio of 3:1 (w/w).

(1987). Removal of starch lipids by various solvents appears to be species-dependent. Extraction with methanol using a Soxhlet apparatus (Schoch 1964a) removed lipids from corn but not wheat starch, whereas boiling 75% ethanol appeared more efficient for wheat than corn starch (Table II). Ninety-five percent ethanol in a Soxhlet (Schoch 1964b) appeared to remove only 75% of starch lipids. Biliaderis et al (1986) cited X-ray and DSC evidence indicating that amylose-lipid complexes do not occur in native starches.

Pasting Curves

Amylograms of prime and low-lipid starches in 1% CMC and in water are given in Figures 5 and 6, respectively. Removal of lipids changed the shape of the amylographs in the heating cycle (Fig. 5). In the low-lipid starches, paste consistency rose sharply as the temperature increased just beyond gelatinization, then consistency increased gradually up to 95° C. In contrast, the native starches showed a restricted increase in consistency after gelatinization, followed by a plateau in the curve at 70–80° C, and then a rapid increase in consistency up to 95° C (Fig. 5). The amylogram for wheat starch in CMC was examined at 7.5% solids, whereas corn was examined at 3.0% solids. Amylograms of corn starch above 3.0% in CMC did not show clearly the two-stage change of paste consistency.

The amylograms in water at 7.5% starch (Fig. 6) showed that removing the lipids eliminated the pasting peak, reduced consistency and setback, and decreased the pasting temperature by approximately 4 and 15° C for corn and wheat starch, respectively. Our amylograms are in agreement with those of Goering et al (1975), but differ from those of Melvin (1979). Melvin removed 0.4–0.5% lipid from wheat and corn starches by slurrying at 70° C with water-saturated *n*-butanol or by Soxhlet extraction using 85% aqueous methanol. Goering et al (1975) removed 0.23–0.38% lipid from barley and corn starches by Soxhlet extraction using 85% aqueous methanol. Goering and co-workers reported that lipid removal gave a somewhat reduced pasting temperature, negligible pasting peak, improved cooking stability, and reduced setback. They attributed those changes to an amylose-lipid complex in the native starches that helped preserve granular structure during the cooking cycle. At high cooking temperature, the complex broke down but reformed during the cooling cycle.

Melvin (1979) reported that lipid-removal from corn and wheat starches reduced the pasting temperature but increased the pasting peak, paste consistency, and setback. Medcalf et al (1968) also found that wheat starch with reduced lipid content (Soxhlet-extracted with methanol) gave a somewhat higher paste consistency in CMC solution than native wheat starch. Lorenz (1976) found that the peak viscosity of wheat starch (92° C) was not affected by prior Soxhlet extraction of the starch with 80% methanol.

These conflicting results may be explained if the lipids were not completely removed from starch during extraction with warm aqueous alcohol, and the remaining lipids formed a complex with amylose during the "defatting" procedure. Amylose complexing agents (with mp in water >90° C) are known to increase pasting temperature, paste consistency, and setback viscosity with wheat

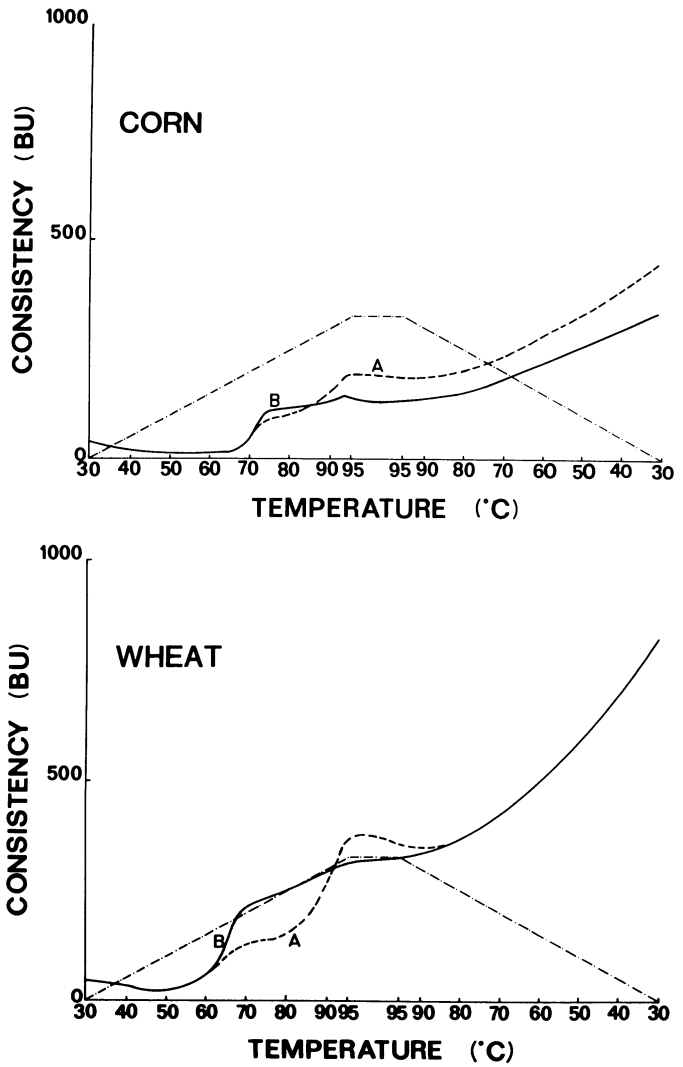

Fig. 5. Amylograms of corn and wheat starch (**A**) and low-lipid corn and low-lipid wheat starch (**B**) in 1% carboxymethylcellulose. BU = Brabender units.

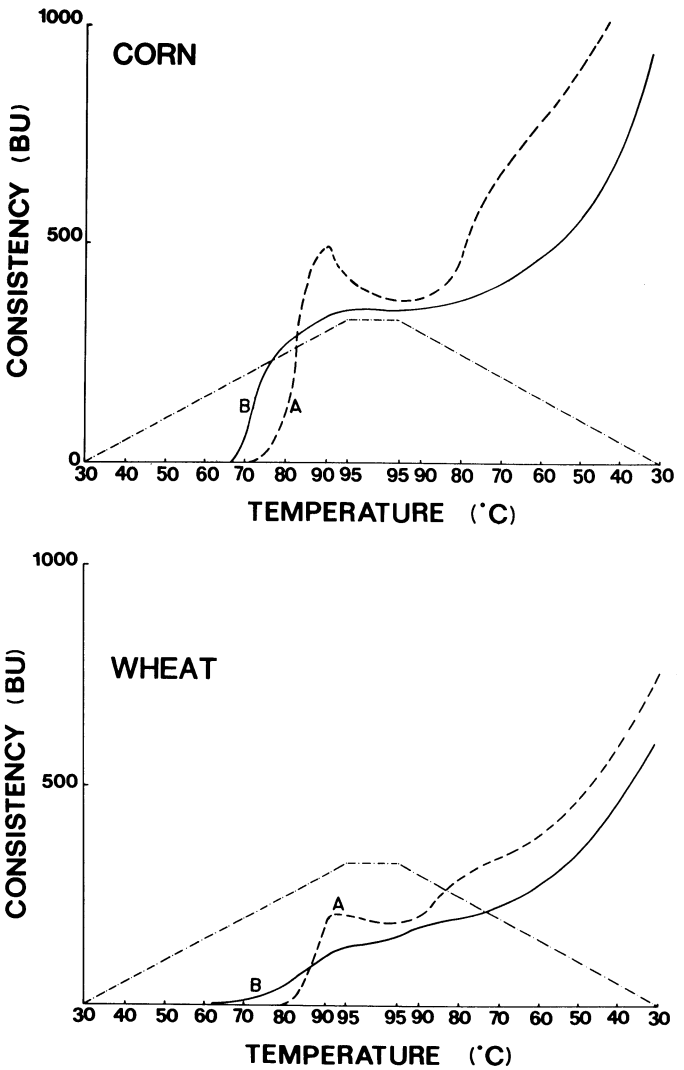

Fig. 6. Amylograms of corn and wheat starch (**A**) and low-lipid corn and low-lipid wheat starch (**B**) in water.

and corn starches (Krog 1973), but to a lesser extent with tapioca starch (Moorthy 1985).

Swelling Power and Solubility

The swelling power and solubility data in Table I for wheat starch are useful in explaining the amylograph curves of both wheat and corn starches. When the low-lipid wheat starch was heated to 75° C, the consistency of the paste rose rapidly (Fig. 5) because of the quick release of approximately 17% starch solubles into the continuous phase of the paste. The solubles were determined to be approximately 75% amylose by IBC (Table I). Continued heating from 75 to 95° C gave a smooth increase in consistency because there was more swelling and approximately 4% more solubles. Upon cooling from 95 to 30° C, the paste consistency also increased smoothly (Fig. 5).

The presence of native lysolecithin (approximately 1%) in the prime wheat starch changed its behavior during pasting. The first rapid rise in consistency of the paste between 55 and 75° C (Fig. 5) was accompanied by a rapid increase in solubles up to approximately 9% of the starch's weight. Continued heating of the prime wheat starch paste between 75 and 85° C gave little change in solubility or swelling power of the starch, which was reflected in the unchanged paste consistency between 70 and 80° C in the amylogram (Fig. 5).

The second rapid rise in the consistency of the prime wheat starch paste occurred from 85 to 95° C (Fig. 5), which coincided with a second rapid release of solubles (+9%) into the continuous phase and increased swelling power of the swollen granules (Table I). It appears that the amylose bound to lysolecithin in the granule started to be released at approximately 85° C in the amylograph, which is the same temperature estimated for the release of amylose from a complex with sodium dodecyl sulfate (Gray and Schoch 1962) and sodium stearoyl 2-lactylate (Ghiasi et al 1982). As mentioned before, Wren and Merryfield (1970) estimated that the 1% lysolecithin in wheat starch is sufficient to satisfy only one fourth of the available lysolecithin-binding capacity (LBC) of the amylose in the starch. Kugimiya and Donovan (1981) used enthalpy measurements in the DSC to determine that amylose has an LBC of 14% by weight. From that value and the amylose (30%) in wheat starch, we can predict that wheat starch has a maximum LBC of 4.2%, which is in agreement with the prediction of Wren and Merryfield (1970).

It is hypothesized that the hot pasting peak in the amylograms (Fig. 6) of prime wheat starch may involve a combination of two phenomena. At the warm temperatures immediately after gelatinization, the amylose-lysolecithin crystals, which form during gelatinization at 50–60° C (Kugimiya and Donovan 1981), are insoluble, and somehow that makes the granules resist swelling. At temperatures greater than 85° C, the amylose-lysolecithin crystals inside the wheat starch granules gradually melt, and this phenomenon allows the lysolecithin to begin to diffuse from the granules. However, even at temperatures above the melting of the V-type crystals, it seems likely that there is an association between lysolecithin and amylose, and probably amylopectin (Evans 1986, Gray and Schoch 1962). This association gives the polymer molecules increased ionic charge, which, in turn, causes increased hydration and swelling of the granules. As the concentration of the surfactant inside the granule decreases because of leaching into the continuous phase at 95° C, the granules lose some swelling power and become more susceptible to shear. Consequently, consistency declines, and the pasting peak decreases. Table I shows that prime wheat starch with 1% native lipid had more swelling power at 95° C than the low-lipid wheat starch.

Surfactants reduce swelling and solubilization of starch in water only as long as they readily complex with amylose (Eliasson 1986) and the complex remains crystalline (Gray and Schoch 1962, Ghiasi et al 1982, Biliaderis et al 1986). Eliasson emphasized that a relatively high concentration of the monomeric species of a surfactant is required to form the amylose-lipid complex. Lysolecithin readily complexes with wheat starch amylose, since it exists in water as micelles that have a high equilibrium concentration of monomeric molecules.

The amylograms for low-lipid corn starch showed the same features as low-lipid wheat starch (Figs. 5 and 6). It is hypothesized that the shape of the pasting curve for prime corn starch was mediated by complexing between amylose and the approximately 6:3 (w/w) mixture of free fatty acids and lysolecithin (Tan and Morrison 1979).

Addition of Wheat Lipids to Wheat Starch

Prime wheat starch was impregnated with 0.5, 1.0, and 2.0% additional lipids isolated from wheat starch. The pasting curves of the starches, which contained totals of approximately 1.5, 2.0, and 3.0% lysolecithin, respectively, are given in Figure 7. The increased pasting peak and consistency of the pastes with added wheat lipid may be explained by formation of additional amounts of lysolecithin-amylose complex inside the starch granule. The high pasting peak seen with 2% added wheat starch lipids together with a second peak at approximately 75° C in the cooling cycle support our explanation for the shape of the amylogram of prime wheat starch. The peak at 75° C during the cooling cycle was especially striking; such a peak had been noted by Osman and Dix (1960) when they heated corn starch with a variety of mono-fatty acid esters. The peak in the cooling cycle probably resulted from the same two phenomena previously described. With cooling, the number of lysolecithin molecules associated with starch molecules increases, and the increasing charge on the molecules increases the swelling of the gel phase. But as the temperature is decreased, at some point, the amylose-lipid complex loses solubility and crystallizes, the granules swell less, and some water of hydration of the molecules may be released into the continuous phase. Those actions reduce paste consistency, and a peak is observed in the cooling cycle. It should be mentioned that adding 2% lysolecithin (based on starch) to the amylograph bowl prior to heating gave almost the same curve as impregnating the starch with 2% wheat starch lipids. Those findings agree with the work of Eliasson (1986). We did not add wheat starch lipids at levels greater than 2% to starch, even though it can be predicted that increasing effects should occur with up to 2.8% added (total of 3.8%) lysolecithin (Kugimiya and Donovan 1981).

If the wheat starch lipids (lysolecithin) were added to the hot paste at 95° C, the leached amylose would complex with lysolecithin outside the granule during cooling. The amylograms in Figure 8 show that increasing the amount of added lysolecithin reduced the peak on the cooling side (at approximately 72° C) much compared to adding the lysolecithin at 30° C prior to cooking (Fig. 7). Moreover, setback was eliminated from the pasting curve upon adding 2% lysolecithin to the hot paste. Figure 9 gives a direct

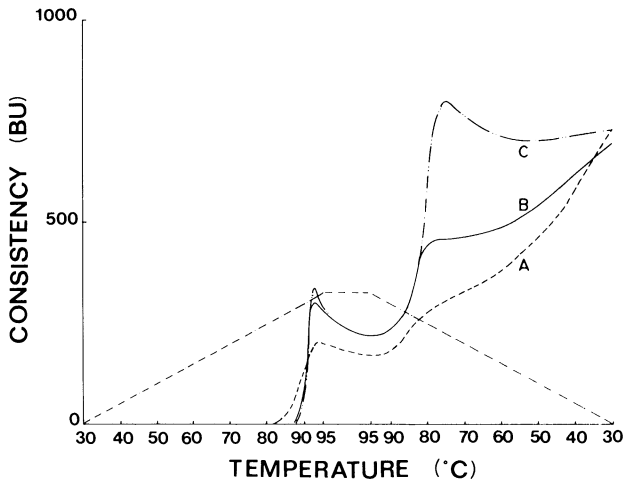

Fig. 7. Amylograms of wheat starch that had been previously impregnated with 0.5, 1.0, and 2.0% additional wheat starch lipids (curves **A**, **B**, and **C**, respectively). The total lipid contents, including the 1% native lipids, were approximately 1.5, 2.0, and 3.0%. All curves were run at 7.5% dry solids. BU = Brabender units.

comparison of paste curves when the wheat lipids (2%) were added before or after cooking. It appears that much of the lysolecithin was prevented from complexing inside the granule, and so the gel phase (granule remnants) did not undergo swelling during cooling. Furthermore, the amylose concentration in the continuous phase declined because of crystallization of the V-complex. The net result was no increase in viscosity during cooling from 60 to 30° C when 2% lysolecithin was added to the amylograph at 95° C (curve D, Fig. 8).

Texture of Gels from Prime Corn and Wheat Starch

In order to obtain precise gel texture measurements by static loading, it is imperative that a gel have a smooth, even surface. To that end, we overfilled a mold with cooked paste, allowed the gel to set, and removed the excess gel with a cheese cutter. Gels with ≤8% solids were prepared from starch that had been cooked to 95° C in the amylograph. An alternate method to obtain smooth surfaces was used on gels containing greater than 10% solids. A CMC-thickened slurry of starch granules was prepared, poured into molds, and then heated in an oven (Maningat 1986). Krusi and Neukom (1984) used precooked starch as a thickener instead of CMC. Maningat (1986) showed that 1% CMC did not exert synergism on the strength of starch gels, a conclusion that we confirmed. The curves in Figure 10 show that adding 1% CMC to differing concentrations of starch increased gel strength by a constant value of approximately 3–4 kP over that of the starch gel in water alone. The relative gel strengths of corn and wheat starches at 5–10% solids were the same with and without 1% CMC, as shown by the similarity of the slopes of the lines in Figure 10.

The firmness of corn and wheat starch gels at 6, 7, and 8% solids was followed with time of aging at 25° C (Fig. 11). Firmness rose rapidly for approximately the first 8 hr, then increased slowly. The rapid firming of a starch gel is explained by the rapid retrogradation of the amylose in the dispersed phase (Miles et al 1985). In more concentrated wheat starch gels (40–55%), Krusi and Neukom (1984) found that the rate of gel firming increased with starch concentration. Furthermore, much of the firming of the gel was due to retrogradation inside the swollen granules. Freshly prepared 40 and 50% wheat starch gels (cooked at 100° C for 110 min and cooled to 20° C) contained 5.4 and 2.6% solubles, respectively, and the water retention capacity of the swollen granular phases decreased during seven days of storage at 20° C.

We found that corn starch gels were more rigid than wheat starch gels below 6% solids (Fig. 11), perhaps because the increased swelling power of the corn starch granules gave a more concentrated solution of amylose in the continuous phase of corn as opposed to wheat starch. At all concentrations between 7 and 30% starch, wheat starch gave a more rigid gel with a higher firmness and breaking point than corn starch (Figs. 11 and 12). Typical load-deformation curves for gels with 8, 20, and 25% solids are shown in Figure 13. These curves consistently showed that wheat starch gels were more elastic and less brittle than corn starch gels above 7% starch solids. It appears that wheat starch gels at high levels of solids contain either a more concentrated solubles phase or a more linear wheat amylose (Takeda et al 1987) in the soluble phase, which increases gel strength.

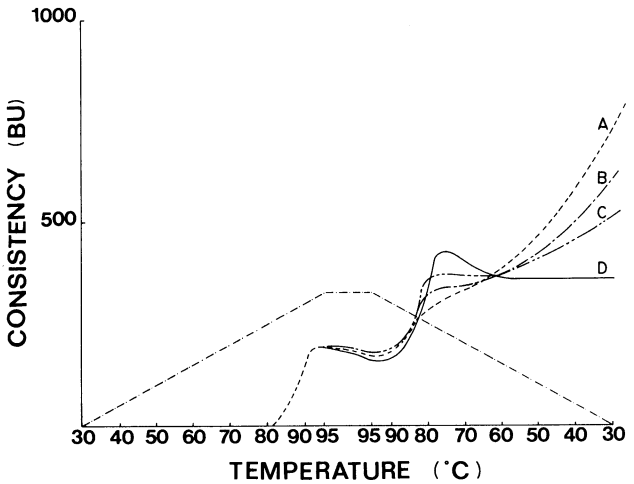

Fig. 8. Amylograms of wheat starch (7.5% solids) cooked to 95° C followed by addition of 0, 0.5, 1.0, and 2.0% wheat starch lipids (curves **A, B, C,** and **D** respectively), followed by cooling. BU = Brabender units.

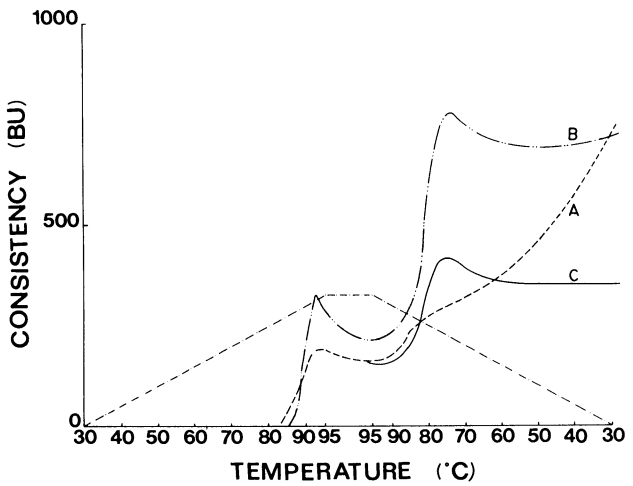

Fig. 9. Amylograms of wheat starch in which 2% wheat starch lipids were added before (**B**) and after (**C**) cooking compared to native wheat starch (**A**). BU = Brabender units.

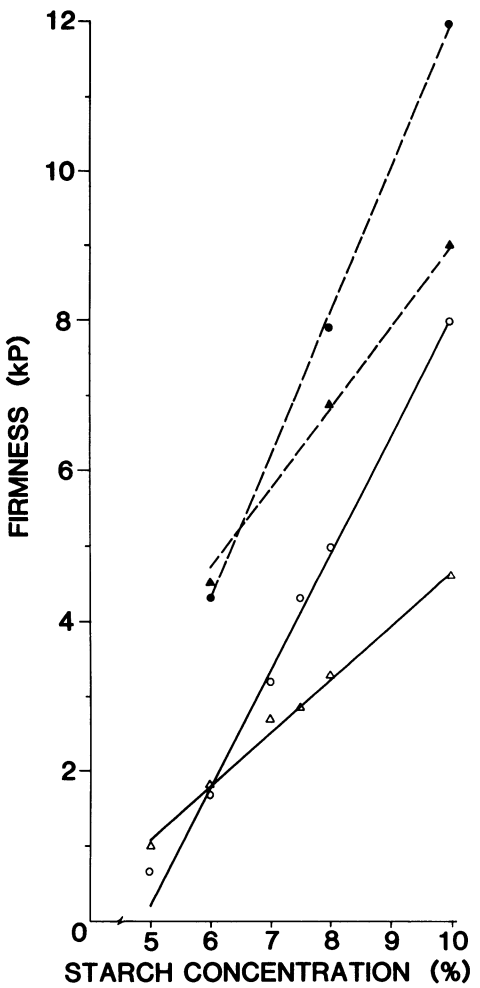

Fig. 10. Firmness (Voland-Stevens) of starch gels made with and without 1% carboxymethylcellulose, (Δ and o, corn and wheat starch, respectively, in water; ▲ and ●, corn and wheat starch, respectively, in 1% carboxymethylcellulose). kP = kilopoise.

Effect of Starch Lipids on Gel Firmness

The naturally occurring lipids in prime corn and wheat starches reduced gel strength in 7.5% gels by approximately 50% (Table III). When wheat starch granules were impregnated with wheat lipids at 1–2% above the native level, cooked in the amylograph, and cooled 24 hr to produce gels, the firmness of the gels was somewhat lower than when the extra lipid was added to the starch already cooked to 95° C in the amylograph. Apparently, keeping the amylose inside the wheat starch granules reduced gel firmness by lowering the concentration of amylose in the continuous phase.

Adding the 2% extra wheat lipid produced wheat starch gels that were almost as soft after 24 hr of aging as those from a hydroxypropylated wheat starch. Even more surprising was the finding that wheat lipids appeared to increase the firmness of gels prepared from the hydroxypropylated wheat starch after 24 hr. The weakest gel after 24 hr was found in the low-lipid hydroxypropylated wheat starch (Table III). However, after storage for several days, the gels of the hydroxypropylated wheat starch were observed visually to become more firm in the absence of wheat starch lipids than in their presence.

TABLE III
Firmness of Starch Gels (7.5% dry solids) Stored at Two Temperatures for 24 hr

Type of Starch Gel	Gel Firmness, kP (grams force) 4°C	25°C
Low-lipid wheat	10.1 (522)	7.0 (360)
Low-lipid corn	9.6 (497)	6.5 (333)
Prime wheat	5.1 (263)	3.9 (203)
Prime corn	3.6 (185)	2.5 (128)
Prime wheat impregnated with		
0.5% Wheat lipids	4.4 (227)	3.1 (161)
1.0% Wheat lipids	1.5 (73)	1.3 (66)
2.0% Wheat lipids	0.7 (37)	0.7 (37)
Prime wheat with lipid (2.0%) added at 30° C before cooking	0.6 (30)	0.5 (28)
Prime wheat with lipid added after cooking to 95° C		
0.5% Wheat lipids	4.1 (209)	2.8 (142)
1.0% Wheat lipids	2.6 (136)	2.0 (104)
2.0% Wheat lipids	1.3 (69)	1.0 (51)
Low-lipid hydroxypropylated wheat	0.6 (32)	0.4 (21)
Hydroxypropylated wheat	1.1 (58)	0.8 (42)
Hydroxypropylated wheat with lipid added after cooking to 95° C		
0.5% Wheat lipid	1.7 (87)	1.2 (64)
1.0% Wheat lipid	1.7 (85)	1.2 (63)

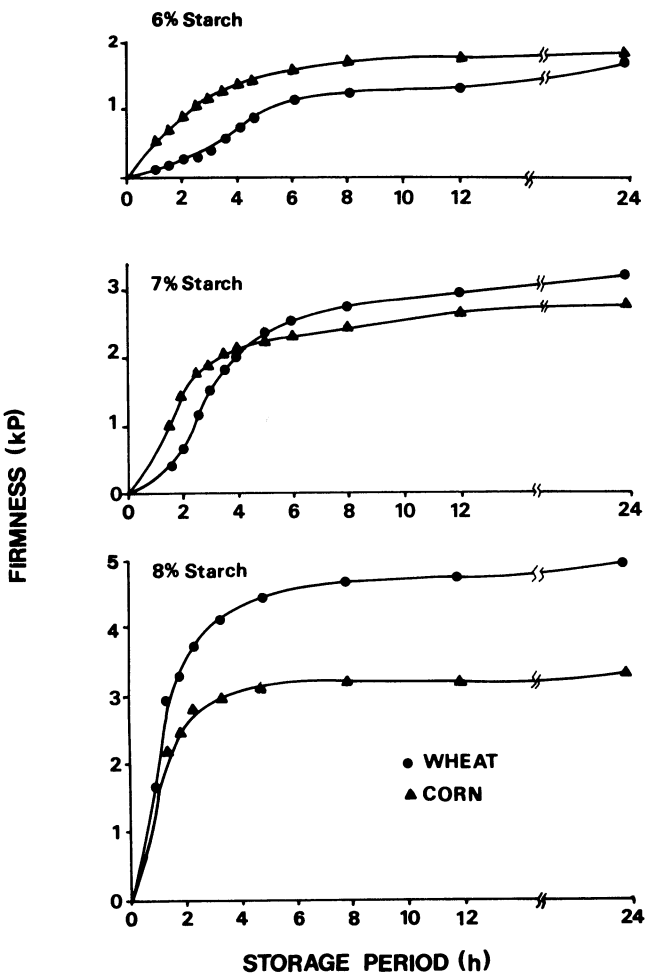

Fig. 11. Firmness (Voland-Stevens) of corn and wheat starch gels after aging 0–24 hr at 25° C. kP = kilopoise.

Stability of Gels

Wheat starch gels at 7.5% solids gave less syneresis during cold

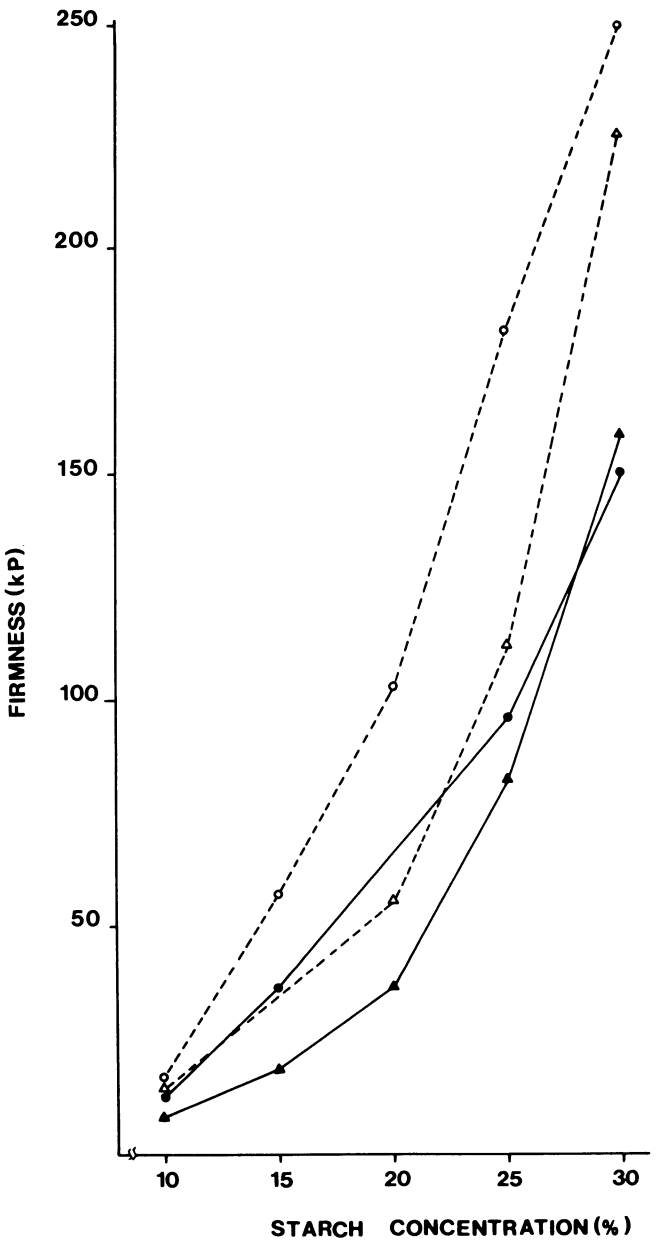

Fig. 12. Firmness (Instron) and yield force of gels made by cooking 10–30% starch in 1% aqueous carboxymethylcellulose and cooling 24 hr at 25° C (Δ, yield force of corn; o, yield force of wheat; ▲, firmness of corn; ●, firmness of wheat).

storage and freeze-thaw treatment than did corn starch gels (Table IV). However, the hydroxypropylated wheat starch was much more stable than either of the unmodified starches.

Adding 2% wheat lipids to the prime wheat starch improved the cold-storage stability of wheat starch gels. Nonetheless, freeze-thaw stability was only marginally affected. The data suggest that lysolecithin can be used to reduce the rate of firming and gelling of wheat starch gels when they are stored above freezing temperature. However, modification of the starch is needed to give stability during freeze-thaw conditions and long-term cold storage.

TABLE IV
Syneresis of Starch Gels (7.5% solids) After Cold Storage and Freeze-Thaw Treatment

Type of Starch Gel	Water Separated After Cold Storage at 4°C, %			Water Separated After Freeze-Thaw (%)		
	3 Days	7 Days	10 Days	1 Cycle	2 Cycles	3 Cycles
Low-lipid wheat	8	11	22	57	64	70
Low-lipid corn	19	22	25	75	75	76
Prime wheat	3	8	16	66	70	73
Prime corn	7	18	20	72	75	76
Prime wheat with lipid added after cooking to 95°C						
0.5% Wheat lipid	12	13	22	54	64	64
1.0% Wheat lipid	15	19	26	56	67	69
2.0% Wheat lipid	5	6	9	54	62	66
Low-lipid hydroxypropylated wheat	0	2	2	0	0	0
Hydroxyproplylated wheat	0	0	0	0	0	0
Hydroxypropylated wheat with lipid added after cooking to 95°C						
0.5% Wheat lipid	0	0	0	0	0	0
1.0% Wheat lipid	0	0	0	0	0	0

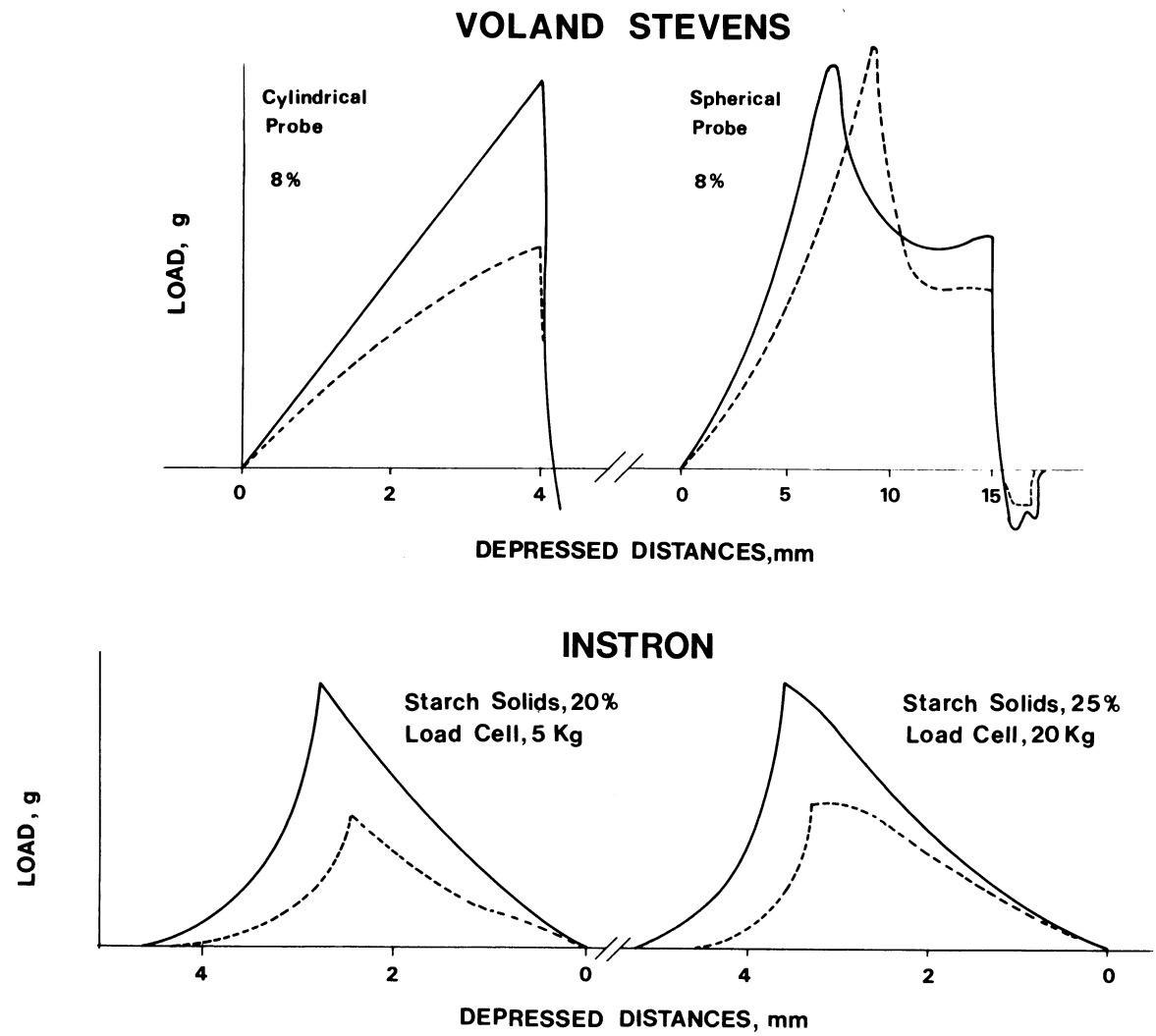

Fig. 13. Force-distance curves (Instron) for 20 and 25% gels made in 1% carboxymethylcellulose and aged 24 hr at 25° C. The yield stress of a gel was calculated from the peak on a curve, and firmness from the load at 2-mm depression. Curves are for corn (-----) and wheat (—). Voland-Stevens curves are shown at the top for gels containing 8% solids.

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