

# A Rheological Investigation of Oat Starch Pastes<sup>1</sup>

J.-L. DOUBLIER,<sup>2</sup> D. PATON,<sup>3</sup> and G. LLAMAS<sup>2</sup>

ABSTRACT

Cereal Chem. 64(1):21-26

Oat starch (variety Sentinel) was subjected to a controlled pasting procedure and subsequently examined for several physicochemical and rheological properties. Oat starch pastes displayed marked thixotropy in their shear stress/shear rate flow curve in contrast to wheat and corn starches pasted at the same concentration. Higher swelling power and solubility values for oat starch gave rise to correspondingly higher values for the volume fraction of the disperse phase,  $\phi$ , and of the concentration of

solubles in the continuous phase,  $C_s$ . Amylose and amylopectin were unexpectedly co-leached from oat starch granules under some measure of influence of the internally bound starch lipids. The implications of these differences in physicochemical properties are discussed in relation to the observed rheological behavior of oat starch pastes. These differences also serve as a possible explanation for oat pastes or gels being more translucent and less susceptible to retrogradation.

Oat starch, isolated from comminuted oat groats by a mild extraction with sodium carbonate at pH 10.0, exhibits pasting behavior distinctly different from wheat and corn starches (Paton 1977, 1979). In particular, the swollen granules are more shear sensitive, and when subjected to cooling, the hot starch paste viscosity has been shown to increase rapidly in the 70–80°C range. Here the paste develops 80–90% of its final viscosity when the temperature has returned to 25°C. Several factors have been suggested as contributors to this behavior, including a higher swelling power and solubility, lower apparent amylose content, and the presence of an intermediate molecular weight fraction (Paton 1979).

Morrison et al (1984) studied the relationship between amylose and lipid content of starches from diploid cereals and found that, in oats, a substantial difference exists between the apparent amylose content and the true amylose content (where amylose determination is made following removal of all lipid material by refluxing the starch in an excess of 3:1 v/v *n*-propanol/H<sub>2</sub>O). Thus, oat starches have a level of amylose comparable to that of other normal cereal starches, a fact which further complicates the explanation for the observed marked differences in past behavior and gel characteristics for this starch.

The present study was undertaken to obtain more fundamental rheological data that might assist in explaining the previously observed oat paste and gel behavior.

## MATERIALS AND METHODS

### Starch Extraction

Oat starch was isolated from the variety sentinel by the method of Paton (1977). Oats were dehulled and cleaned before grinding in an impact mill to pass a 0.5-mm screen. Starch was extracted from the ground groats by forming a slurry in water (1:10, solid to liquid), adjusting the pH to 10.0 with a 20% sodium carbonate solution, and stirring at a temperature of 45°C (two 30-min extractions). Centrifugation was carried out in 250-ml screw-capped plastic bottles at 5,000 × *g* for 30 min. The resuspended aqueous slurry was passed over a 0.06-mm screen to remove coarse bran, and the starch milk was further centrifuged at 1,500 × *g* for 30 min. A layer of fine bran and protein that still remained on top of the starch was removed with a spatula. The residual starch was resuspended in water neutralized with 2*N* HCl, centrifuged at 1,500 × *g* (30 min), and rewashed with distilled water before freeze-drying. Wheat and corn starches studied were of industrial origin

and were obtained from Roquette Frères (F-62136-Lestrem, France).

### Analytical

Oat starch, isolated by the above procedure, had a protein content of 0.5% and a free lipid content of 0.3% as determined by standard Kjeldal N and AOAC methods (AOAC 1984). A factor of N × 6.25 was used for protein.

Iodine affinity was measured by the method of Larson et al (1953) on native and defatted starch.

Starch was defatted by the procedure of Morrison et al (1981) using 20 volumes of an *n*-propanol and water (3:1 v/v) mixture in screw-capped vials in a boiling water bath (two 2-hr + one 1-hr extraction).

### Pasting Procedure

Starch pastes were prepared according to the standard procedure described by Doublier (1981). Weighed amounts of starch were slurried in 200 ml of water then rapidly heated in a round bottom jacketed glass vessel equipped with a variable speed anchor-shaped stirrer. Because it has been observed during previous studies that the final rheological behavior of starch pastes is dependent upon the conditions used in paste preparation (Doublier 1981), starches were pasted at combinations of both a high (750 rpm) and low (200 rpm) speed and under a high (6°C/min) and low (1°C/min) heating rate. Upon reaching 96°C, the pastes were held at that temperature for a further 30 min to ensure proper cook. Actual final starch concentrations, used for subsequent rheological measurements, were determined by drying aliquots of the cooked starch pastes overnight at 103°C.

Pasting of native and defatted oat starch was also performed by an alternate procedure using the Ottawa starch viscometer (Voisey et al 1977), but no rheological evaluations were conducted on pastes cooked by this method.

### Swelling and Solubility Measurements

Swelling power and solubility of starch pastes were determined on 0.4% (w/w) starch dispersions according to the method of Leach et al (1959). The amount of the soluble material in the supernatant was estimated from its volume and concentrations by the orcinol method as described by Tollier and Robin (1979). The swelling power was determined by weighing the swollen sediment and correcting for solubles. Characterization of the supernatants was achieved by their spectral behavior in iodine; the sediment was similarly examined following solubilization with 1*N* KOH.

### Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) of native and defatted oat starch was performed using a Dupont 910 cell base connected to a Dupont 1090 analyzer. Starch was mixed with sufficient water in a vial to give a 50–55% moisture paste and the vial was then capped and stored at 4°C overnight. After allowing the vial to come to room temperature, sufficient moisture-equilibrated starch

<sup>1</sup>Presented in part at the 70th AACC meeting, Orlando, FL, September 1985. Contribution no. 695 of the Food Research Centre, Agriculture Canada.

<sup>2</sup>Inra Laboratoire de Physicochimie des Macromolécules, 44072 Nantes Cedex France.

<sup>3</sup>Food Research Centre, Agriculture Canada, Research Branch, Ottawa Ontario K1A 0C6 Canada.

(3–5 mg dry basis) was withdrawn, weighed into a pan, and tamped down with a fine glass rod to ensure even packing. The pan was then sealed and reweighed several times over a 30-min period to ensure that no leaks had occurred during a lid closure. Using purified silica sand in the reference pan, samples were analyzed at a heating rate of 10°C/min over the temperature range 40–140°C. Samples were then cooled to 40°C and rerun.

### Rheological Measurements

Cooked starch pastes were removed from the glass cooking vessel at about 85°C and rapidly transferred to the measuring cup of a Rheomat 30 concentric cylinder viscometer. The measuring system had the following specifications: internal radius, 22.9 mm; external radius, 24.2 mm; height, 56.5 mm. The measuring assembly was thermostatted at 70°C, and shear stress ( $\sigma$ ) versus shear rate ( $\gamma$ ) curves were developed automatically over a programmed shear rate range from zero to 600 s<sup>-1</sup> and by return to zero. In most cases a second run was performed in order to assess the presence or absence of thixotropy. In some cases, measurements were carried out at 100 s<sup>-1</sup> after this second run, and the apparent viscosity at this shear rate was calculated from the ratio ( $\sigma/\gamma$ ).

## RESULTS AND DISCUSSION

Analytical data pertinent to the discussion of rheology of oat starch pastes was obtained from the oat variety Sentinel: iodine-binding capacity (IBC) at 25°C = 2.10%; apparent amylose content (based on an amylose IBC of 18.8%) = 11.2%; lipid content (*n*-propanol/H<sub>2</sub>O, 3:1 at 95°C) = 1.3%; IBC of lipid-free starch (25°C) = 5.06; amylose content (based on an amylose IBC of 18.8%) = 26.8%. It is clear that the internal starch lipid content (1.3%) is considerably higher than the free extractable lipid value (0.3%). This internally bound lipid also alters the value obtained for amylose content. It was estimated that approximately 60% of the amylose present was inaccessible to iodine in native oat starch granules (Table I).

**TABLE I**  
Effect of Complete Lipid Removal on Iodine Binding Capacity (at 25°C) of Oat and Corn Starch

Starch	% Iodine Binding Capacity		% Complexed Amylose
	Native	Lipid Free	
Oat	2.1	5.06	60.0
Corn	3.6	5.4	35.0

**TABLE II**  
Swelling Power (G) and Solubility (S) of Oat Starch Under High or Low Heating Rate and Low Shear Conditions

Temperature (°C)	Supernatant		Sediment	
	S (%)	$\lambda$ max	G (g/g)	$\lambda$ max
High heating rate				
60	0.46	...	3.68	582
70	0.86	570	6.95	582
80	1.67	595	8.54	585
90	4.44	595	15.2	587
95	23.7	590	30.8	575
96				
0 min	28.6	587	35.2	565
5 min	33.9	585	38.5	555
10 min	34.0	585	40.5	560
20 min	37.2	585	45.2	560
Low heating rate <sup>a</sup>				
70	1.12	580	7.92	590
80	1.32	590	7.42	595
90	3.74	595	14.65	590
95	17.5	585	21.7	585
96				
0 min	22.7	585	26.1	560
5 min	43.5	583	41.0	552
20 min	...	...	...	...

<sup>a</sup>0.4% Starch dispersions.

The swelling power and solubility patterns of oat starch are presented in Table II under high heating rate-low shear (HR-LS) and low heating rate-low shear (LR-LS) conditions. Very little difference was found between the two procedures in terms of values obtained. The most notable aspect of these data were the values for the  $\lambda$  max of the supernatant and sediment phases. The absorbance of the supernatant phase never exceeded 600 nm. This is in marked contrast to results previously obtained for wheat starch. During leaching, when the wheat starch solubles content approaches the true amylose content of the starch (i.e., 27–28%), the  $\lambda$  max of the supernatant phase was within the range 635–645 nm. As the amount of solubles increases, the value for the  $\lambda$  max decreases (Doublie 1981). This indicates that, for normal cereal starch, amylose is preferentially leached and that amylopectin can only begin to solubilize once the amylose has leached. For oat starch, both amylose and amylopectin are co-leached throughout the pasting process.

The oat and wheat sediment phases are similar, both showing a tendency towards enrichment of amylopectin ( $\lambda$  max = 560 nm). Table III compares the swelling power and solubility at 96°C (after a 20-min holding period) for oat, wheat, and corn starches pasted under the HR-LS and LR-LS procedures. Whereas corn and wheat show variability in swelling power and solubility according to the preparation procedure, oat starch is much less sensitive, while exhibiting much higher swelling power and solubility. Figures 1 and 2 represent complete curves and show the progressive development for both parameters under the pasting conditions employed. The flow curves for different concentrations of oat starch pastes are shown in Figure 3 for the HR-LS condition only. It is evident that above a concentration of 2.0%, oat starch displays strong thixotropic behavior that increases with starch concentration. Figure 3 also shows the presence of a phenomenon known as stress-overshoot which immediately follows the yield point. All of the curves show a decline from this overshoot with increasing shear rate up to about 175–200 s<sup>-1</sup>. This behavior is quite similar to that of most viscoelastic polymers determined over time at steady state measurement under a constant shear rate. The classical explanation for stress-overshoot is the formation, at rest, of a weak structural network that must then be overcome and broken down following start up of the viscometer (De Kee et al 1983). This weak network may be more or less disrupted when transferring the paste from the cooking vessel to the viscometer because of limited, but unavoidable, shear effects. Therefore, the first part of the “up” curve can only be taken as a qualitative indication of the possible internal structure of the oat starch paste, because the magnitude cannot be estimated with any degree of accuracy. Above a shear rate of 100 s<sup>-1</sup>, however, good reproducibility was observed. As shown in Figure 3, a strong dependence on concentration was also observed; the apparent viscosity at the maximum shear rate was eight times higher for a 4.3% oat starch paste than for the 2.2% paste.

Figure 4 shows a comparison at 3.4% concentration between oat, wheat, and maize starches pasted by the HR-LS condition. The shape of the up curve of the 3.4% paste in Figure 4 is somewhat different than that in Figure 3 (3.35% concentration). This likely relates to the difficulties just mentioned for the measurement of

**TABLE III**  
Comparison of Swelling Power (G) and Solubility (S) for Maize, Wheat, and Oat Starches<sup>a</sup>

Preparation Procedure	Starch Type	S (%)	G (g/g)
High heating rate low shear	Maize	40.0	33.3
	Wheat	26.7	24.1
	Oat	37.2	45.2
Low heating rate low shear	Maize	27.0	25.5
	Wheat	37.4	15.5
	Oat	45.9	46.4

<sup>a</sup>0.4% Starch dispersions at 96°C (20 min).

stress-overshoot. The marked differences between the three starches are quite evident. Maize and wheat display pseudoplastic flow, although a very slight thixotropy is noticed. The curves designated by the number 2 represent the second run following the completion of the first curve. For oat, the first run destroyed most of the structure associated with the paste but thixotropic flow is still visible. A third run would result in almost complete disappearance of the area between the up and down curves. Maize and wheat starches showed little difference between curves of their first and second runs and less evidence of a yield point. Oat starch, on the other hand, still shows an apparent yield point even after the second shearing run, indicating a structural difference in this starch paste. Measuring the thixotropic flow under well-controlled conditions is thus a second means of estimating the amount of internal structure of such suspensions. The comparison of the three starches in this respect indicates that the structural network of hot oat starch pastes is stronger than that of either wheat or corn when assessed at the same concentration. A similar ranking of flow curves was obtained when all three starches were pasted by the different combinations of two heating and stirring rates. These results are similar to those reported by Paton (1977, 1979) using the Ottawa starch viscometer, where oat starch displayed a higher hot paste consistency and a dramatic consistency increase on cooling compared to similar paste concentrations of wheat and corn starches. It is noteworthy that the stronger internal structural network of hot oat starch pastes is further magnified upon cooling and appears not to be influenced by the steady state shear rate.

#### Interpretation of Viscometric Measurements

Cooked starch pastes may be considered as suspensions of swollen particles dispersed in a macromolecular viscous matrix. Both phases contribute to the overall rheological behavior of the paste. It is therefore advantageous to determine the relative contribution of each phase to final paste rheology. Doublier (1984) published a short study wherein an estimate of both phases was calculated from a knowledge of the swelling power and solubility data.

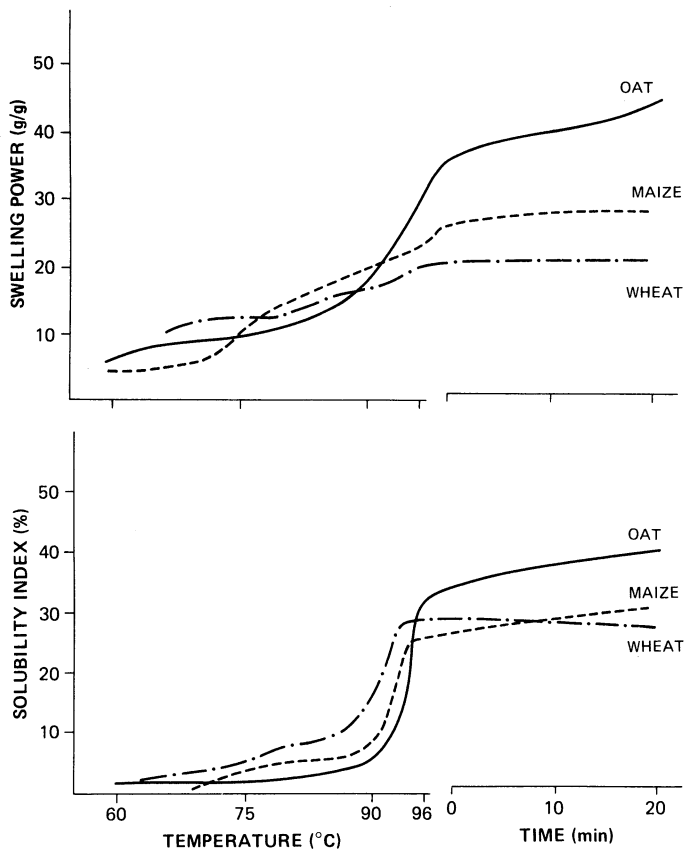


Fig. 1. Swelling power and solubility patterns for oat, maize, and wheat starches cooked under high heating rate and low shear conditions.

The volume fraction of the sediment or discontinuous phase ( $\phi$ ) may be expressed as

$$\phi = (1 - S/100) \cdot C \cdot G \quad (1)$$

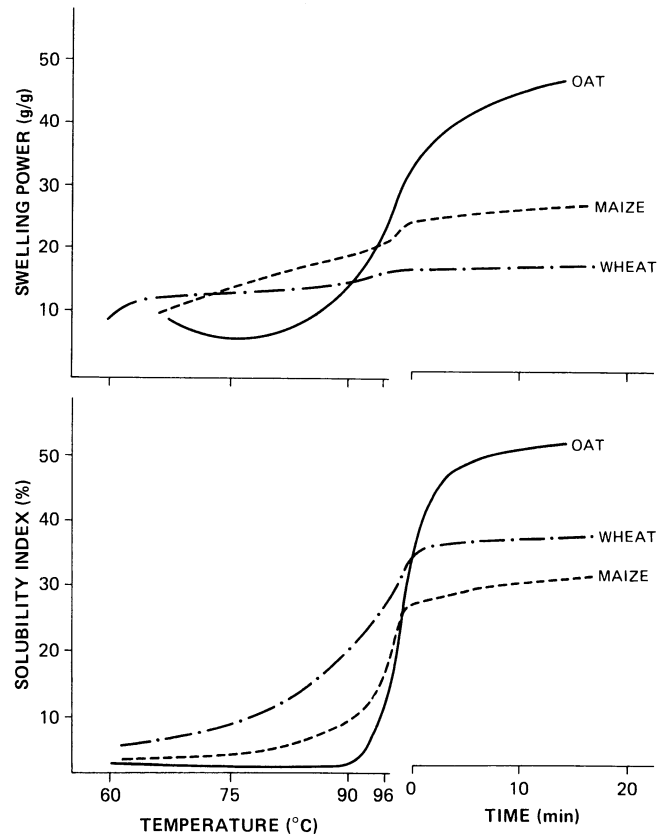


Fig. 2. Swelling power and solubility patterns for oat, maize, and wheat starches cooked under low heating rate and low shear conditions.

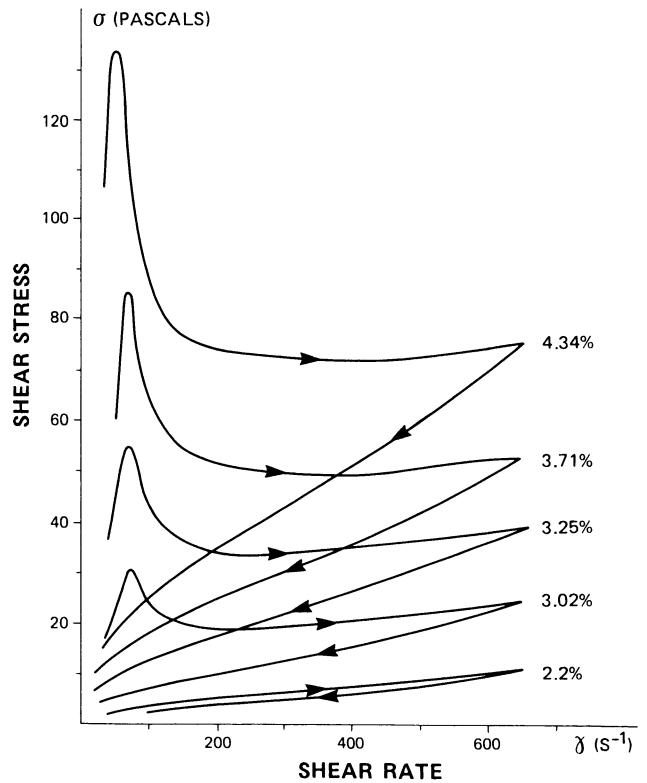


Fig. 3. Flow curves of oat starch pastes at different concentrations. Pasting conditions: high heating rate and low shear. Measuring temperature: 70°C.

and the concentration of starch in the continuous phase,  $C_s$ , as

$$C_s = 1/(1 - \phi) \cdot C \cdot S \quad (2)$$

where  $S$  = solubility (g/100 g),  $C$  = starch concentration (%), and  $G$  = swelling power (g/g).

Equations 1 and 2 were used to calculate values for  $\phi$  and  $C_s$  for oat, wheat, and corn starches following pasting at 96°C and cooling to 70°C under the HR-LS and LR-LS conditions. Results are shown in Tables IV and V. From this data, the volume fraction ( $\phi$ ) for oat starch is 1.3 and 2.5 times higher than for maize and wheat starches respectively for the LR-LS cooking conditions and 1.4 and 1.6 higher respectively for the HR-LS condition. However, the corresponding values for  $C_s$  (oat) are 4.2 and 5.2 times higher than those for maize and wheat (LR-LS) and 7.0 and 14.4 times higher for the HR-LS treatment.

Figure 5 is an illustration of how  $\phi$  and  $C_s$  vary with starch concentration;  $\phi$  varied linearly with concentration, whereas  $C_s$  was exponentially related to concentration. Several relationships exist that express the variations of the relative viscosity as a function of the volume fraction of the dispersed phase. Quemada (1978) proposed the following equation:

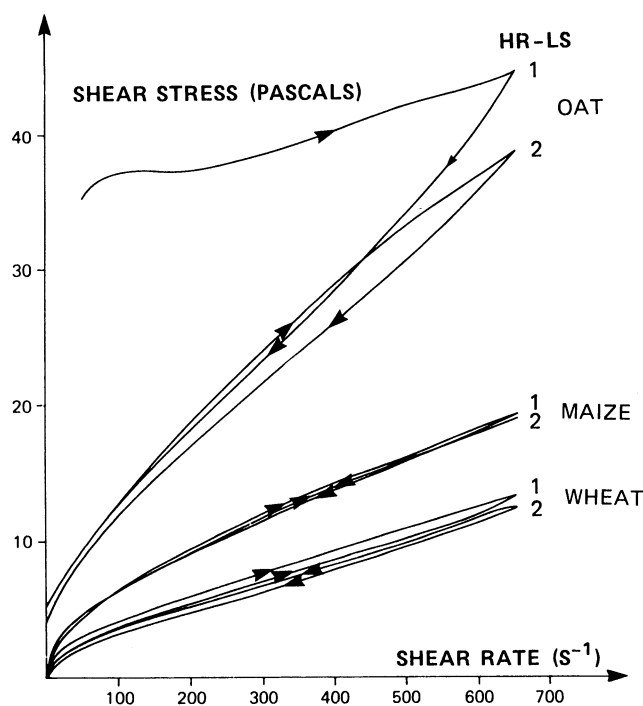
$$\eta_{rel} = \eta_s/\eta_c = F(\phi) = 1 - 0.5k\phi \quad (3)$$

where  $\eta_s$  = viscosity of the suspension,  $\eta_c$  = viscosity of the

**TABLE IV**  
Estimation of  $\phi$  and  $C_s$  and Comparison with Viscosity Measured at 70°C After Pasting<sup>a</sup>

Preparation Procedure	Starch	$\phi$	$C_s$	$\eta_{100 \text{ s}^{-1}}$ (mPa·s)
High heating rate low shear	Maize	0.68	4.24	61.7
	Wheat	0.60	2.28	33.1
	Oat	0.96	33.00	107.0
Low heating rate low shear	Maize	0.63	2.36	29.5
	Wheat	0.33	1.90	6.46
	Oat	0.84	9.79	128.8

<sup>a</sup>Starch concentration 3.4%.



**Fig. 4.** A comparison of flow curves of oat, maize and wheat starch pastes: concentration, 3.4%; pasting conditions, high heating rate and low shear; 1 = first run, 2 = second run.

continuous phase, and  $k$  = intrinsic viscosity of the suspension.

It was shown in a previous study that this equation fitted experimental data for wheat and corn starch pastes, providing that the viscosity of the continuous phase was low and that  $\phi$  did not exceed 0.5 (Doublier 1984). The value for  $\phi$  depends upon the botanical origin of the starch, the pasting procedure used, and the shear rate employed during pasting. It should also be emphasized that  $\phi$  depends upon the concentration of the continuous phase ( $C_s$ ).

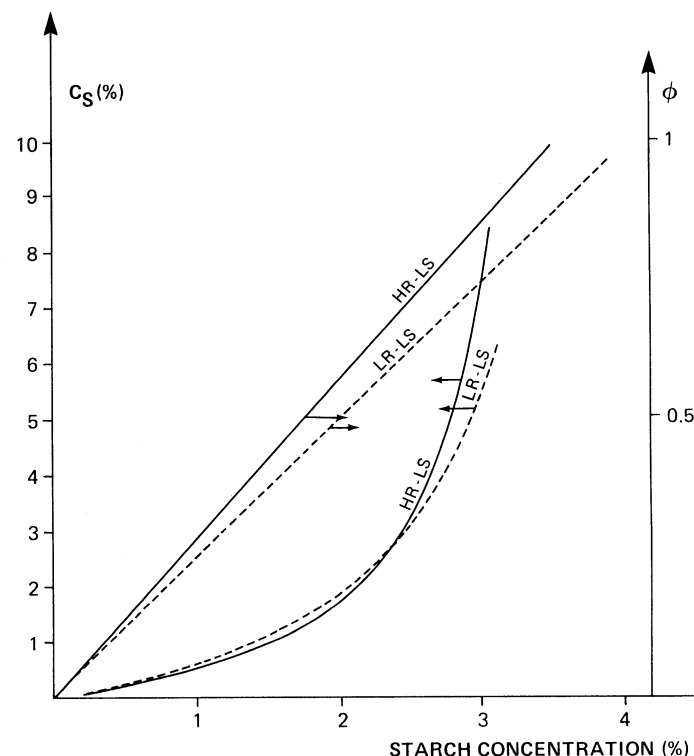
Obviously equation 3 is more valid at low starch concentration. In the results reported here, the starch concentration was much higher (3.4%) but was selected so as to correspond to the concentration used for actual rheological measurements. Because  $C_s$  varies exponentially with concentration, the values of 9.79 and 33.0 for oat starch are likely to be unreliable because they are on the steep "up" slope of the  $C_s$  versus concentration curve (Fig. 5), and  $\phi$  values are close to unity. This is also borne out by the apparent viscosity calculated at the 100 s<sup>-1</sup> shear rate.

Table V shows that  $\eta$  (100 s<sup>-1</sup>) values for oat starch are of the same magnitude, whereas the  $C_s$  values are not. Overall, however,  $\eta_{100 \text{ s}^{-1}}$  values are significantly different from those calculated for wheat and maize, and these differences can be qualitatively related to variations in  $\phi$  and  $C_s$ .

Table V shows the effect of completely removing the internally bound oat starch lipids by the *n*-propanol and water (3:1%) method of Morrison et al (1984). Lipid removal resulted in a marked reduction of swelling power and an increase in solubility. These changes were accompanied by an increase in the  $\lambda_{max}$  of the

**TABLE V**  
Effect of Complete Lipid Removal on Swelling Power, Solubility and Spectral Behavior of Oat Starch

Type of Starch	95°C		
	Swelling Power (g/g)	Solubility (%)	$\lambda_{max}$ (solubles) (nm)
Native oat starch	46.3	33.3	598
Lipid-free oat starch	17.2	50.7	626



**Fig. 5.** Volume fraction,  $\phi$ , and concentration of solubles in the continuous phase,  $C_s$ , as a function of concentration of oat starch. HR-LS = high heating rate and low shear; LR-LS = low heating rate and low shear conditions.

starch-iodine complex of the supernatant solubles. Lipid removal resulted in oat starch having a swelling power, solubility, and nature of solubles quite similar to corn and wheat. The shift in  $\lambda_{max}$  to a higher value suggests that lipid removal allows amylose to leach selectively from the oat granules. This is in contrast to the results of Table II, which support the contention that, for native oat starch granules, swelling proceeds in a manner that results in the co-leaching of amylose and amylopectin.

Figure 6 shows a DSC scan of native oat starch; the existence of a strong amylose-lipid complex is evident. The rerun scan shows the reversibility of this complex formation and melting, whereas the lower temperature endotherm (associated with the melting of starch crystallinities and the resultant phenomenon of gelatinization) was irreversible. Figure 7 shows the effect of removing the internally bound starch lipids. The first run curve shows a much reduced low temperature endotherm that was likely the result of some measure of crystallite annealing because of the reflux procedure during the removal of lipid. The main feature of the initial and the rerun curves is the absence of any starch-lipid endotherm.

Internal lipid-free oat starch was pasted in the Ottawa starch viscometer and the curve compared with native oat starch (Fig. 8). The lipid-free starch swells earlier and over a broader range caused by the refluxing regime and an earlier solubilization of amylose. This annealing also produces a lower consistency of hot paste, which may be related to a reduced swelling capacity for this starch. The key effect of lipid removal is seen on the cooling portion of the pasting curve where it more closely resembles corn and wheat starches. At the end of the pasting cycle, the defatted starch was poured into a beaker and examined 1 hr later. This starch formed an opaque rigid gel that retrograded rapidly when stored in a refrigerator at 4°C for 48 hr. The native starch exhibited a semitranslucent, loose, and adhesive gel that did not change after 48 hr of storage at 4°C.

Although not reported here, experiments were also performed where oat lipids were added back to extracted oat and wheat starches and the products pasted. The pasting curves were quite different from those of the native and defatted starches, having very low set backs not unlike those usually observed for waxy maize starch. The pastes were opaque but did not gel, remaining instead as salves that continued to be pourable after 48 hr of storage at 4°C. These add-back experiments were not able to reproduce the pasting curve for native oat starch. This is not surprising, as the mechanism of binding sites for added-back lipid is likely to be quite different and produce a different type of starch-lipid binding than would occur in the native granule as a result of contact between the internal lipids and starch chains as the granule undergoes swelling and pastings.

Several conclusions may be drawn from the results presented in this study. From the rheological point of view, oat starch differs widely from other cereal starches, displaying shear stress/shear rate flow curves that exhibit marked thixotropy. This indicates the

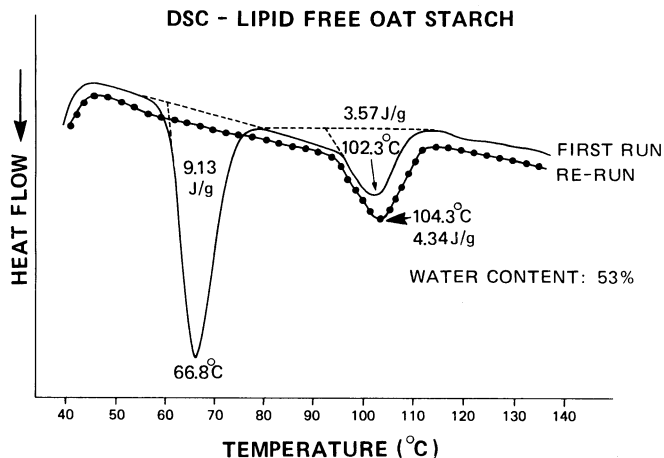


Fig. 6. Differential scanning calorimetry thermograms of oat starch.

existence of a stronger structural network within the paste than is found for wheat and corn starches. Oat starch also has a higher swelling power and solubility, which in turn give rise to higher values for the volume fraction of the dispersal phase and for the concentration of solubles in the continuous phase. Because of the higher swelling power, there is less liquid available to the soluble portion of the starch. Both these parameters likely contribute to raising the viscosity of the entire cooked dispersion. In addition, the nature of the solubles in oat starch is such that both amylose (alone or in complexed form) and amylopectin are co-leached from the native granule. This is in marked contrast to other cereal starches where amylose is preferentially leached despite the high internal starch lipid content of these starches (Morrison et al 1984). Removal of internal oat starch lipids gives an extracted starch paste that has cold gelling properties similar to native wheat and

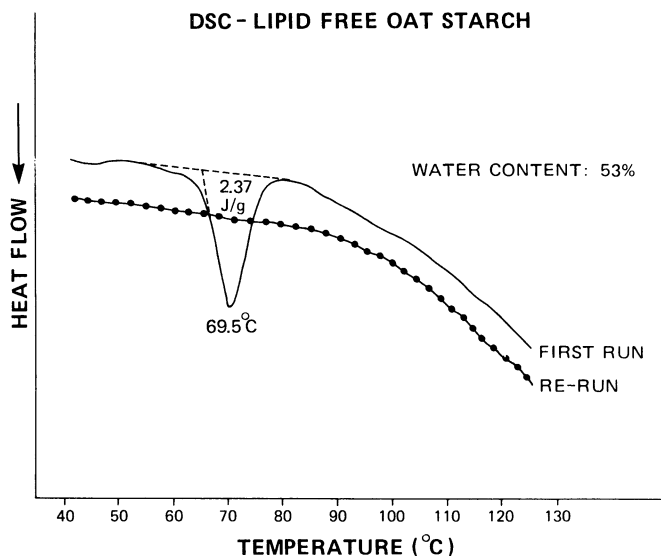


Fig. 7. Differential scanning calorimetry thermograms of oat starch defatted by the procedure of Morrison et al (1984).

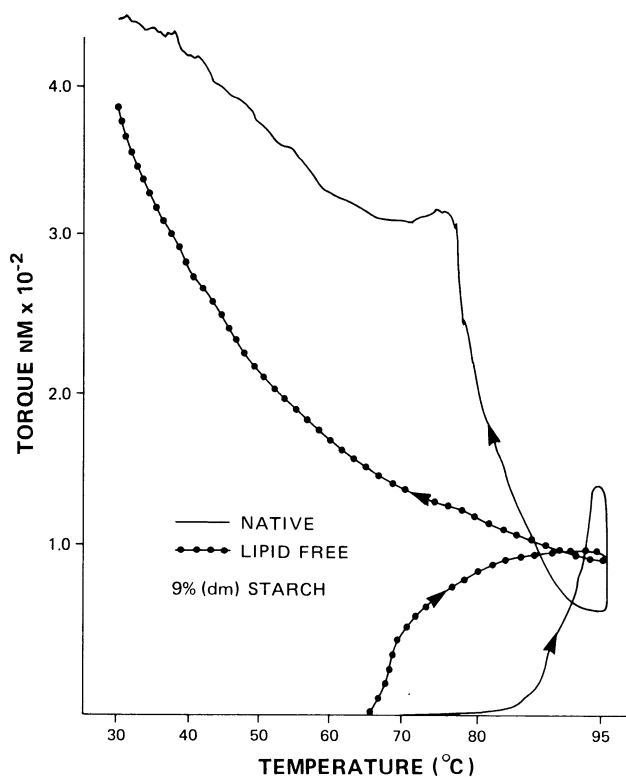


Fig. 8. Pasting curves of native and defatted oat starch performed with the Ottawa starch viscometer (Voisey et al 1977).

corn starches. In this case, amylose is preferentially leached from the granules and starch swelling is restricted. It may be inferred that a relationship exists between starch swelling and opacity; the less the swelling power, the more opaque the paste or gel. Similarly, rigidity of starch gels may result from the phase separation of linear and branched starch species, the former promoting a more rapid retrogradation. In the case of native oat starch, the simultaneous leaching of linear and branched starch species could be expected to retard the recrystallization of the linear amylose and increase the storage stability of refrigerated starch pastes. Further, the high swelling capacity of oat starch would result in a more translucent gel.

The actual mechanism of the involvement of internally bound lipids on the pasting and rheology of oat starch pastes and gels remains unexplained at present. One might have expected oat starch to have a very low swelling power because of the higher amount of lipid present. Starch-bound lipids thus seem to play quite a different role in oat starch than in wheat or corn.

It is possible that the spatial location of lipids within oat starch granules in relation to the disposition of amylose and amylopectin is also important in governing the leaching of amylose and amylopectin from this starch. It would be very useful to be able to locate these sites of starch-bound lipid deposition, and future studies are planned with this objective in mind.

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[Received June 25, 1986. Accepted September 15, 1986.]