

CHANGES IN AMYLOID CARBOHYDRATES DURING PREPARATION OF SWEET POTATO FLAKES

ABSTRACT

Precooked dehydrated sweet potato flakes were prepared by the enzyme activation technique. After lye peeling and grinding, the puree was held at 75°C for timed intervals, then processed into flakes. Flakes from each time period were analyzed for maltose, dextrin and starch content; for changes in starch and dextrin molecular size; and for changes in rheological properties. It was found that all of these factors changed most rapidly during the first 10 min of conversion and that significant starch hydrolysis occurred during lye peeling. A slow but steady change took place among the macromolecular components for 60 min of conversion time. Our data suggested that the properties of macromolecular components could be used as a guide in the selection of optimum conversion conditions.

INTRODUCTION

PRECOOKED DEHYDRATED sweet potato flakes are manufactured by a process which employs the "enzyme activation technique" as described by Hoover (1967). In this technique, puree prepared from raw sweet potatoes is rapidly heated to about 75°C and held at that temperature until the naturally occurring amylolytic enzyme system has converted enough starch into other amyloid polymers and maltose to produce an acceptable texture. Overconversion of the starch yields a product with an undesirably low viscosity while underconversion yields a thin, lacy flake.

Carbohydrate changes occurring in sweet potatoes during enzymatic conversion of the starch have been studied by several groups, but their investigations have been confined to measuring the mono- and disaccharides. Hoover and Harmon (1967) found that maltose is the only sugar produced and its production is 90% complete after 10 min at 75°C; yet Hoover (1967) found that quality changes still occurred after 45 min conversion at 75°C. Deobald et al. (1968; 1969) reported that maltose content provided an estimate of flake quality. However, they indicated that α -amylase activity measurements of the raw roots provided a much more reliable guide to conversion time necessary for production of high quality flakes.

The amylolytic enzyme system of sweet potatoes has been shown to consist mainly of β -amylase (Balls et al., 1948) and a smaller amount of α -amylase (Ikemiya and Deobald, 1966). Beta-amylase hydrolyzes starch and starch fragments to maltose. The primary mode of action of α -amylase is to hydrolyze starch by an endo process into amyloid polymers called dextrans. Starch and dextrans are classified as gums (Glicksman, 1974). When these are present in food, they strongly influence textural and organoleptic properties by virtue of high water-binding capacity and their interaction with one another as well as other food constituents. Sweet potatoes contain from 16–25% starch (fresh basis) and during the conversion process large amounts of starch are available for hydrolysis by α -amylase. Thus it appeared to us that amyloid carbohydrates other than maltose may strongly influence flake quality. No information was available either on the dextrans or

on subtle changes which the starch undergoes during the conversion step of the flake manufacturing process. This study relates the length of time the sweet potato puree was held at the conversion temperature of 75°C to changes in the amyloid components of the finished flakes.

MATERIALS & METHODS

JEWEL VARIETY (USDA Grade No. 1) sweet potatoes obtained immediately after harvest were stored at 15°C. After 3 days the roots were washed, peeled in 10% lye for 5–6 min (104°C), trimmed and pureed in a comminutor fitted with a 0.03-in. screen. A small batch of the puree was then passed through a steam injector, which instantly raised its temperature to 103°C. This portion served as the zero time sample in which no enzymatic conversion occurred. The remainder of the puree was processed (Hoover, 1967) by using the steam injector to instantaneously raise the temperature to 75°C. Batches of the puree were placed in an incubator and held at 75°C for 10, 20, 30, 60 and 120 min periods. After the desired time interval, each puree batch was reheated in the steam injector to 103°C to stop further enzyme activity. After cooling, each batch of puree was then processed into flakes on an 8 × 10 in. double drum dryer heated with steam at 60 psig. The moisture level of each flake batch was calculated from the moisture loss occurring when the flakes were dried to constant weight at 80°C in vacuo. Each batch of flakes was analyzed for sugar (glucose, reducing sugar and maltose content), dextrin and starch levels. Molecular size changes of amyloid components were determined by changes in the starch and dextrin blue values and in the intrinsic viscosities. Rheological properties of the flakes were determined by apparent viscosity after reconstitution of the flakes with water.

To measure the effect of the peeling method on the conversion process, a lot of the freshly dug roots was divided into two groups. One group was hand peeled, pureed and immediately heated to 103°C in the steam injector to minimize any possibility of enzyme action. In an effort to measure the effect of heat on degradation of the starch, the hot puree was collected and stored in a dewar flask which had been preheated to 100°C. Samples for sugar analysis were removed from the raw unheated puree and from puree in the dewar flask after 20 min and 60 min. The second group of potatoes was lye peeled, pureed and held at room temperature to measure the effect of lye peeling. Samples for sugar analyses were taken immediately after pureeing and after 45 min at ambient temperature.

Carbohydrate analysis

Sugars. Duplicate samples of flakes or puree were weighed, mixed with 14 ml boiling water, allowed to stand for 1 hr and then made to 100 ml with ethanol. After equilibrium (ca 7 days), reducing sugar content (Hodge, 1964) and the total alcohol soluble carbohydrate (Dubois et al., 1956) were measured on appropriate dilutions. The glucose content was measured by the glucose oxidase procedure of Dekker and Richards (1971), except that glucose oxidase free of carbohydrate hydrolases was used. Maltose levels were calculated by subtracting the glucose concentration from the reducing sugar concentration.

Total dextrin extract (TDE). Duplicate weighed flake samples were exhaustively extracted with 80% ethanol to remove sugars and then with ether and methanol to remove lipids. This sugar-free material was then extracted with 90:10 water-ethanol (Walter et al., 1975). The supernatant which contained the total dextrin extract (TDE) was divided into two portions. One portion was analyzed for total carbohydrate concentration with the phenol-sulfuric acid procedure (Dubois

et al., 1956) and for dextrin concentration by the enzymatic procedure of Dekker and Richards (1971). The other portion of the TDE was freeze dried and used for intrinsic viscosity measurements. The insoluble residue remaining after the dextrans had been extracted was dried and used for starch determination.

Starch. Starch was quantitated by subjecting dried, dextrin-free solids to the amyloglucosidase-glucose oxidase procedure (Dekker and Richards, 1971). When purified starch samples were needed for intrinsic viscosity measurements, a different preparation procedure was used. Duplicate 5.0g samples of dextrin-free solids from each time period were extracted with 100 ml of 90% dimethylsulfoxide (DMSO) on a rotary shaker for 10 hr. The solvent was removed after centrifugation and the residue extracted two more times with fresh solvent. The supernatants were combined, treated with three volumes of ethanol-methanol (1:1) and the precipitate collected by centrifugation. The precipitate was purified by two additional dissolution-precipitation treatments. After the final centrifugation, the pellet was heated with *n*-butanol at 100°C for 3 hr and centrifuged again. It was then removed, dissolved in 100 ml boiling water and shaken 1.5 hr in a flask containing 100 ml toluene. The aqueous layer was removed and the starch was precipitated by adding 4 volumes of an ethanol-methanol (1:1) mixture. The starch samples were washed with ethanol, dried at 50°C in vacuo for 24 hr and stored in a desiccator until intrinsic viscosity and blue value measurements were made.

Rheological measurements

Viscosity determination. Flakes were reconstituted by adding boiling water (5 parts water to 1 part flakes), stirring for 1.0 min, then capping the sample and allowing it to cool for 3 hr before taking measurements. A Haake Rotovisko model R V-1 rotary viscometer was used for rheological evaluation of the reconstituted flakes. An SVP-II sample cell designed specifically to minimize separation of suspended particles was used. This cell consists of a cylindrical rotor which is attached to the Rotovisko by a rotating shaft and is mounted inside a stationary cylinder of slightly larger diameter. The sample to be examined occupies the gap between the rotor and the fixed surface. The apparent viscosity in centipoise (η) is obtained by determining the amount of resistance to revolution the rotor encounters due to the sample. Each sample in duplicate was tested (23°C) at each of 10 rotor speeds, ranging from 3.6 rpm to 583.2 rpm.

Enzyme analysis

Alpha and Beta amylase. The enzyme activities were measured on the freshly harvested roots before processing. Three composite samples, each consisting of tissue from three roots, were prepared and the activities of α and β -amylase were determined on the expressed juice (Walter et al., 1975).

Physical analysis

Blue value. Iodine binding capacity of each starch and dextrin sample was expressed as the blue value (BV). The pH of a solution of the carbohydrate was adjusted to 6.5 and then an aliquot pipetted into a 100 ml volumetric flask. The blue color was developed with acidic potassium iodide-iodate solution (Wolf et al., 1970). The absorbance was measured at 680 nm on a Cary Model 15 spectrophotometer. The glucose content of a separate aliquot was measured as described (Dekker and Richards, 1971). From these data the blue value was calculated (McCready and Hassid, 1943).

Intrinsic viscosity. Flow times for intrinsic viscosity calculations were made with #50 semi-micro Cannon-Ubbelohde dilution viscometers (Cannon Instruments, State College, Pa.). For starch, 0.5M NaOH was used as solvent and for TDE 0.1M KCl was used as solvent. The sample was dissolved and filtered. A 1.0-ml portion was charged into a calibrated viscometer placed in a water bath at $25.1 \pm 0.05^\circ\text{C}$, and temperature equilibrated. The flow time was then measured in triplicate. A measured amount of solvent was added, mixed, temperature equilibrated and the flow time again measured in triplicate. This procedure was continued until a fourfold dilution was obtained. The diluted sample was removed and the carbohydrate content measured using the phenol-sulfuric acid reagent (Dubois et al., 1956). Carbohydrate concentration for each dilution was calculated. The intrinsic viscosity $[\eta]$ was obtained from these data by using the expression below:

$$[\eta] = \lim_{C \rightarrow 0} [\eta_{sp}/C]$$

To calculate $[\eta]$, a plot of η_{sp}/C vs C was extrapolated to "zero" concentration. Here η_{sp} is the specific viscosity and C is the carbohydrate concentration in grams per milliliter (Greenwood, 1964).

Viscosities for all samples were measured on solutions containing 0.25–1.0% carbohydrate.

RESULTS & DISCUSSION

FOR THE PURPOSE of this discussion, 0 time flakes are those prepared by drum drying a batch of sweet potato puree which had been heated in a steam injector to 103°C. Ten min flakes are those prepared from puree which had been rapidly heated to 75°C, held for a conversion time of 10 min and then rapidly heated to 103°C. Similarly 30, 60 and 120 min flakes are those batches prepared by conversion at 75°C for 30, 60 and 120 min and then heating to 103°C before drum drying.

Carbohydrate compositional changes

Flakes prepared from the 0 conversion time puree contained 12.5% maltose and 7% dextrin (Fig. 1). This result was surprising because instantaneous elevation of the puree temperature to 103°C in the steam injector should have permitted only minimal amyolytic enzyme activity. Sweet potato starch gelatinizes at 68–73°C and α and β -amylases are unable to attack ungelatinized starch. In this process enzymes should have had the proper substrate available for less than 1 sec before being heat denatured. Thus maltose and dextrin production should have been small.

The large amount of maltose present in the 0 time flakes caused us to determine what effect the peeling method might have upon the conversion processes. Consequently, conversion in puree prepared from hand-peeled potatoes was compared to that in puree prepared from lye peeled roots (Table 1). Maltose content of unheated, hand-peeled, raw puree was found to be 0.1% (dry basis) and was increased only to 0.6% by steam injection to 103°C. Moreover, the maltose level of this steam injected puree changed little when it was held 60 min in a dewar flask at 90–95°C. In this case, the maltose level was only slightly increased by the steam injection step reflecting slight amyolytic activity. In contrast, lye peeled raw puree contained 15.6% maltose (dry basis) which increased to 18.7% after being held 45 min at room temperature. We concluded that lye peeling was responsible for the maltose and dextrin levels in the 0 time sample (Fig. 1).

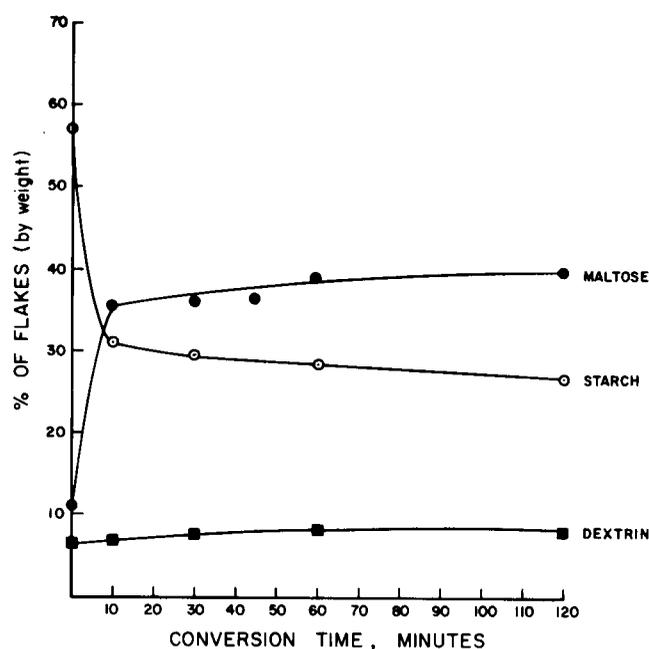


Fig. 1—Changes in concentrations of maltose, starch and dextrin during conversion at 75°C.

Table 1—The effect of peeling method on the formation of maltose in sweet potato puree

Treatment	Maltose as % of dry matter
Hand peeled	
Raw puree	
0 time	0.1
Steam injection to 103°C and held in heated dewar flask	
20 min	0.7
60 min	0.6
Lye peeled	
Raw puree	
0 time	15.6
45 min	18.7

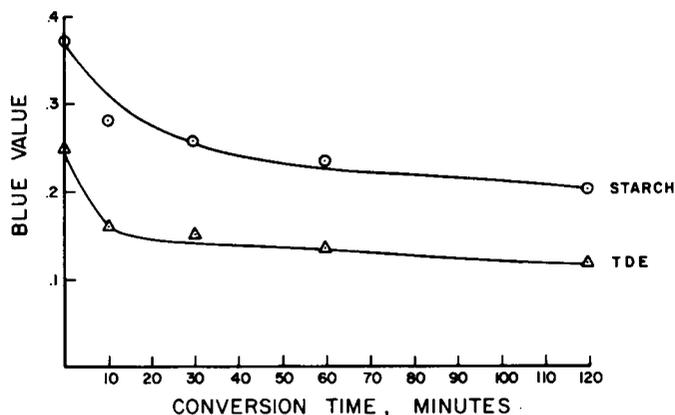
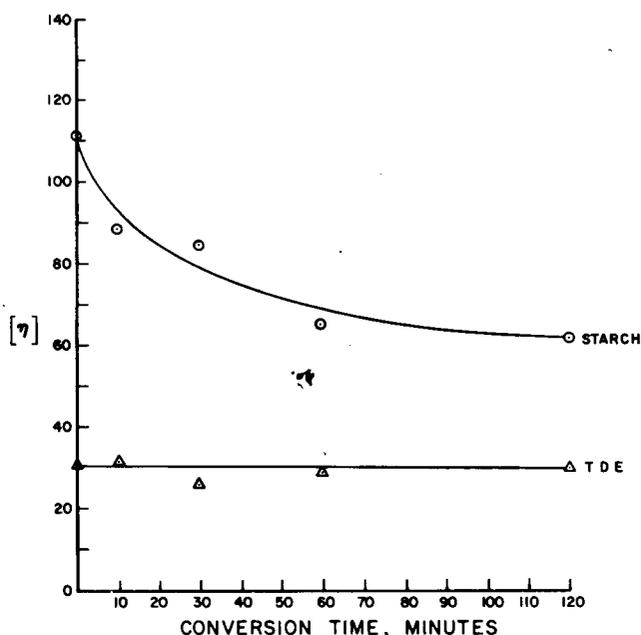


Fig. 2—Blue values for starch and total dextrin extract (TDE) during conversion at 75°C.

Fig. 3—Intrinsic viscosity ($[\eta]$) for starch and total dextrin extract (TDE) during conversion at 75°C.

The lye peeling process is designed to provide sufficient heat and alkali to cause lye-contacted tissue to slough off. The intact potatoes remain in contact with 104°C, 10% NaOH solution for about 5–6 min. Upon removal from the lye bath, the skin and all water soluble surface material were eliminated by tumbling the roots in a strong water spray. A transverse section of a lye-peeled root showed a 5–8 mm deep ring of a different color than other tissue. Microscopic examination of the ring revealed that the starch granules had been ruptured, apparently by heat. Very little maltose was formed in hand-peeled puree which was steam injected to 103°C (Table 1), thus eliminating heat alone as the causative agent. We concluded that amylolytic enzymes from the unheated interior of the root rapidly attacked the gelatinized starch from the “cooked” ring when the lye peeled roots were pureed in the comminutor. The effect of lye peeling upon the conversion process must be taken into account if quality flakes are to be produced.

Although the maltose level in the 0 time flakes made from lye-peeled roots was 12.5%, it rapidly increased and reached a value of 36.5% in those flakes which had been produced by a 10 min conversion period (Fig. 1). After 10 min conversion, the maltose concentration increased only slightly and reached a value of 39% in 120 min flakes. These data are similar to those of Deobald et al. (1969) and Hoover and Harmon (1967). Most dextrin formation occurred (Fig. 1) during the lye peeling step as is evident by the fact that the 0 time flakes contained 7.5% dextrin. Flakes produced by a conversion time of 120 min contained only 8.5% dextrin.

In 10 min flakes (Fig. 1), the starch content decreased from that of 0 time flakes reflecting rapid hydrolysis into maltose and then decreased more slowly. The rapid starch hydrolysis apparently resulted from the combined activities of α and β -amylase. The α -amylase activity of the raw roots was 0.33 APA units/ml juice/min (Walter and Purcell, 1973) and β -amylase activity was 808 mg maltose/ml juice/min. The decreased rate of maltose formation and starch hydrolysis after 10 min was probably due to heat inactivation of the β -amylase which is rapidly denatured at 75°C (Balls et al., 1948). Subsequent changes in amyloid composition were probably due to the action of α -amylase which is stable at 75°C (Ikemiya and Deobald, 1968).

Starch and Dextrin molecular size

Changes in configuration and molecular size of starch and dextrin during conversion are indicated by the blue value (BV) and intrinsic viscosity ($[\eta]$). The blue value, which is the measure of the ability of the molecule to bind iodine, is dependent upon the length of the unbranched chains (Swanson, 1948). Reduction of the molecular size through hydrolysis of amylose, amylopectin or dextrin would be indicated by a BV decrease. Blue values of both starch and dextrin decreased rapidly during the first 10 min of conversion and then more slowly (Fig. 2), indicating that the average linear chain length was reduced by amylolytic enzymes. Blue values were consistently greater for starch than for dextrin.

Starch $[\eta]$ decreased sharply for the first 60 min of the conversion and the rate then slackened (Fig. 3). TDE $[\eta]$ was unchanged. Since $[\eta]$ is related to polymer molecular size, the starch was apparently degraded but TDE was not. However, decrease in BV (Fig. 2) indicated that both TDE and starch were degraded. Walter et al. (1975) showed that the TDE contains other polymers (e.g., pectins). Possibly the molecular size of these other polymers was not changed during conversion and was of such magnitude that changes in dextrin size were masked and no change in $[\eta]$ was observed. The BV, which is a specific measure of the length of linear α -1,4-glucosidic chains, would respond to changes in length of these chains only and would not be affected by the presence of other polymers as would $[\eta]$. Therefore, although the $[\eta]$ values did

not change, we believe that dextrin molecular size decreased as the length of the conversion period increased.

Decrease in molecular size of starch (Fig. 3) continued until 60 min of conversion, while maltose formation slowed after 10 min. This is indicative of change occurring at the macromolecular level which, because of water binding properties of these polymers, strongly influenced the properties of the resulting flakes. Deobald et al. (1969) and Hoover and Harmon (1968) found that significant changes occurred in the bulk density and organoleptic properties of flakes for some time after the accumulation of maltose had stopped. We believe that changes in macromolecular components are responsible.

Rheology of reconstituted flakes

The apparent viscosity of reconstituted flakes (Fig. 4) was found to be dependent upon the amount of time the puree was held at 75°C before flaking. The apparent viscosity changed most between 0 time and 10 min flakes. However, a loss in apparent viscosity also was observed between 10 min and 30 min flakes and between 30 min and 60 min flakes. These changes in apparent viscosity of reconstituted flakes with respect to length of the conversion period are similar to changes in $[\eta]$ of starch isolated from each batch of flakes (Fig. 3) and to changes in the amount of starch present in each batch (Fig. 1). The loss of viscosity reflected a decrease in water-binding capacity of the flakes caused by hydrolysis of the starch into maltose, dextrans and other starch fragments.

The flow behavior of reconstituted flakes was similar to that observed for baked sweet potato (Rao et al., 1975), in that both were found to exhibit pseudoplastic behavior which is characterized by the property of a fluid to become thinner when stirred rapidly (shear thinning). The viscosities which we have reported (Fig. 4) are termed apparent because reconstituted flakes are non-Newtonian fluids (Glicksman, 1969), in that the viscosity changes with respect to the rotor speed of

the viscometer. We have reported the results (Fig. 4) from three rotor speeds but similarly shaped curves were obtained from all 10 speeds.

For 0 time flakes there were large differences in apparent viscosity with respect to rotor speed. For 60 min flakes, however, the differences had diminished. The apparent viscosity of 0 time flakes decreased 34,000 cps between rotor speeds of 3.6 and 10.8 rpm (Fig. 4). The change in apparent viscosity for 10 min flakes, using the same rotor speeds, was 6000 cps. A reasonable explanation is that as starch fragmentation proceeded, the fluid became more ideal. Apparently no starch hydrolysis occurred between 60 and 120 min flakes, since the apparent viscosities at each rotor speed are the same at both 60 and 120 min.

SUMMARY & CONCLUSIONS

THIS STUDY has shown that significant starch hydrolysis occurred during lye peeling. Moreover, maltose production was 90% complete by the end of the first 10 min of the sweet potato flake conversion process following lye peeling. However, changes in macromolecular properties such as starch and dextrin molecular size, as indicated by changes in $[\eta]$ and BV and in the apparent viscosity of reconstituted flakes, occurred until the conversion had been in progress for 60 min. The macromolecular components of flakes are able to bind water and thus strongly influence texture of the reconstituted flakes. The length of the conversion period determines their water-binding characteristics. Since textural properties of flakes change after most maltose production has ceased, it appears that changes in macromolecular components play a role in this change. A method based on macromolecular properties such as the blue value or puree viscosity could be used as a guide to control the conversion process.

REFERENCES

- Balls, A.K., Walden, M.K. and Thompson, R.R. 1948. A crystalline β -amylase from sweet potatoes. *J. Biol. Chem.* 173: 9.
- Dekker, R.F.H. and Richards, G.N. 1971. Determination of starch in plant material. *J. Sci. Food Agric.* 22: 441.
- Deobald, H.J., Hasling, V.C., Catalano, E.A. and McLemore, T.A. 1969. Relationship of sugar formation and sweet potato alpha amylase activity during processing for flake production. *Food Technol.* 23(6): 118.
- Deobald, H.J., McLemore, T.A., Hasling, V.C. and Catalano, E.A. 1968. Control of sweet potato α -amylase for producing optimum quality precooked dehydrated flakes. *Food Technol.* 22(5): 93.
- Dubois, M., Gilles, K.A., Hamilton, J.K., Rebert, P.A. and Smith, F. 1956. Colorimetric method for determination of sugars and related substances. *Anal. Chem.* 28: 350.
- Glicksman, M. 1969. "Gum Technology in the Food Industry," p. 15. Academic Press, New York.
- Greenwood, C.T. 1964. "Methods in Carbohydrate Chemistry," Vol 4, p. 179. Ed. Whistler, R.J. Academic Press, New York.
- Hodge, J.E. 1964. "Methods in Carbohydrate Chemistry," Vol 1, p. 386. Ed. Whistler, R.J. Academic Press, New York.
- Hoover, M.W. 1967. An enzyme activation process for producing sweet potato flakes. *Food Technol.* 21: 322.
- Hoover, M.W. and Harmon, S.J. 1967. Carbohydrate changes in sweet potato flakes made by the enzyme activation technique. *Food Technol.* 21(11): 115.
- Ikemiya, M. and Deobald, H.J. 1966. New characteristic alpha amylase in sweet potatoes. *J. Agric. Food Chem.* 14: 237.
- McCready, R.M. and Hassid, W.Z. 1943. The separation and quantitative estimation of amylose and amylopectin in potato starch. *J. Amer. Chem. Soc.* 65: 1154.
- Rao, V.N.M., Hamann, D.D. and Humphries, E.G. 1975. Apparent viscosity as a measure of moist mouthfeel of sweet potatoes. *J. Food Sci.* 40: 97.
- Swanson, M.A. 1948. Studies on the structure of polysaccharides. 4. Relation of the iodine color to the structure. *J. Biol. Chem.* 172: 825.
- Walter, W.M. Jr., Purcell, A.E. and Nelson, A.M. 1975. Effects of amylolytic enzymes on "moistness" and carbohydrate changes of baked sweet potato cultivars. *J. Food Sci.* 40: 793.
- Wolf, M.J., Melvin, E.H., Garcia, W.J., Dimler, R.J. and Kwolek, W.F. 1970. Amylase determination in dimethyl sulfoxide extracts of maize. *Cereal Chem.* 47: 437.
- Ms received 3/4/76; accepted 4/12/76.

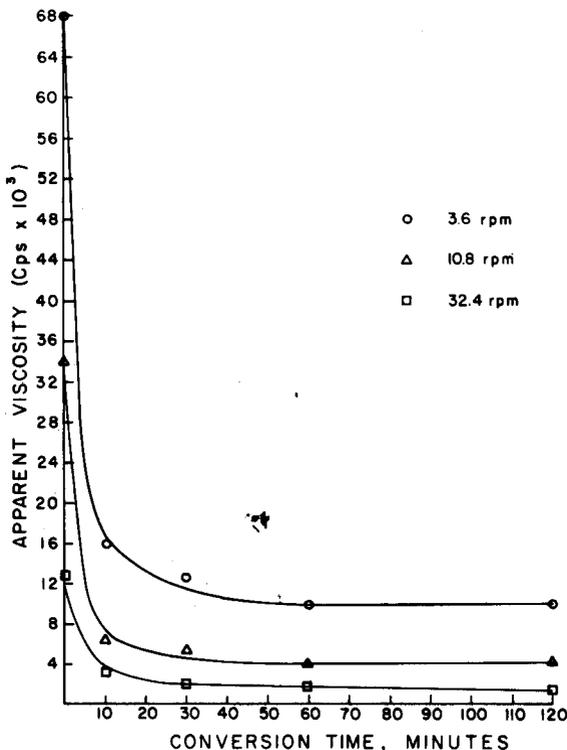


Fig. 4—Apparent viscosity in centipoises (cps) of reconstituted flakes at three rates of shear relative to puree conversion time at 75°C.

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