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ENGINEERING AND PROCESSING

Extrusion Processing Conditions for Amylose-Lipid Complexing[1]

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ABSTRACT

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Normal corn starch containing 25% amylose was extruded with and without stearic acid and at various combinations of barrel temperatures, screw speeds, and moisture contents. The presence and amount of starch-stearic acid complexing was measured using iodine binding capacity, apparent amylose content (iodine spectra of soluble fraction of extrudates in 0.5*N* KOH), and differential scanning calorimetry. Maximum complexing was observed at 110–140°C barrel temperatures, 140 rpm screw speed, and 19% moisture content. Physical properties of extruded starches were also evaluated. The addition of stearic acid before extrusion decreased expansion ratio and water solubility index and increased bulk density.

High-temperature extrusion cooking is used extensively by many food industries to produce various food products with unique texture and flavor characteristics. Desirable properties in the end product are obtained by varying the processing conditions as well as the composition of the raw material. It is recognized that the addition of ingredients such as lipids, proteins, sugar, and salt alter the physical and chemical properties of the extruded foods. Changes in the properties of starchy foods caused by the addition of lipids are attributed to the formation of complexes between amylose and lipids. Researchers have known about the formation of these complexes during traditional cooking processes (like breadmaking) for a long time. Furthermore, complexing has been reported to increase product shelf life. Extrusion cooking of lipid-containing products also results in the formation of these complexes (Mercier et al 1980, Colonna and Mercier 1983, Stute and Konieckey-Janda 1983, Schweizer et al 1986).

Research on extrusion of starches and lipids has been primarily devoted to studying the characteristics of the extrudates. Unfortunately, the manner in which these changes occur has not been as highly researched. Most studies on the effects of processing conditions on lipid binding have been conducted with twin-screw extruders (Mercier et al 1980, Colonna and Mercier 1983, Stute and Konieckey-Janda 1983, Schweizer et al 1986, Galloway et al 1989, Guzman et al 1992). As a result, very little is known about effect of processing variables on complex formation during single-screw extrusion cooking. Twin-screw extrusion studies have been conducted with cereal flours and grits that contain protein, which also binds lipids. Hence, the results reported cannot be applied directly to foods containing varying percentages of starch. Therefore, the objectives of this study were: to optimize the extrusion processing variables of barrel temperature, screw speed, and moisture content of the feed material for maximum complexing of starch with lipids; and to study the interrelationships and propose the mechanism for complexing that would lead to greater understanding of the effects of extrusion of starches with lipids.

To achieve these goals, the research was divided into two parts. The first part consisted of single-screw extrusion of normal corn starch with and without the addition of stearic acid. Processing conditions were: temperatures of 110–170°C, screw speeds of 110–170 rpm, and feed moisture contents of 19–25%. Comparison was made using a 3×3×3 full-factorial design. The degree of lipid binding was determined using: 1) differential scanning calorimetry, 2) changes in starch iodine-binding capacity (IBC) and apparent amylose content, and 3) wavelength of maximum absorbance for starch.

The second part of the research was a study of the effect of lipid binding on the expansion ratio, bulk density, and water solubility index of the starch-lipid extrudates.

MATERIALS AND METHODS

Materials

Commercially available 25% amylose corn starch was received gratis from American Maize-Products Co. (Hammond, IN). Stearic acid (C18:0) was received gratis from Humko Chemical Division of Witco Corp. (Memphis, TN). A 4% lipid substitution level, based on dry weight of starch, was used for all samples.

To obtain better flow of the material into the extruder, the starch powder was granulated before extrusion. Granulated starch samples were mixed with distilled water in a Hobart mixer (model C-100) to adjust the moisture content to desired levels and were stored in plastic jars overnight before extruding.

Extrusion Process

A laboratory extruder (model 2802, C.W. Brabender, South Hackensack, NJ) with a 1.9-cm barrel diameter and a barrel

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length-to-diameter ratio of 20:1. The extruder screw had a compression ratio of 3:1. The temperatures of the die and compression sections of the extruder were varied according to the experimental design. The feed section was held at 70°C. The extruder was fed full while varying the screw speed. The torque input to the extruder shaft was recorded using a torque transducer (model 450A, BLH Electronics Inc., Waltham, MA).

The variables of specific mechanical energy and mass flow rate were monitored using standard procedures (Mercier and Feillet 1975). Specific mechanical energy was calculated by dividing the net power input to the screw by the extrudate flow rate corrected for the loss of moisture at the die. Net power input to the extruder was determined from the torque and angular velocity measurements. For physico-chemical analyses, extrudates were dried at 40°C and ground in a micro-mill (Powdertec 3090, Tecator, Inc., Germany) to pass through a 0.5-mm sieve. The ground samples were defatted in a fat extractor (Soxtec, Tecator) using petroleum ether (bp 34.6°C) to remove uncomplexed lipids.

Iodine Binding Capacity

The iodimetric method of amylose content determination of Schoch (1964) was used to determine the iodine binding capacity of the native and extruded starches. The only modification was that samples were defatted with petroleum ether to remove free lipids before analysis. The bound lipids were not removed and residual iodine binding capacities were determined.

Apparent Amylose Content

Apparent amylose content (iodine blue value) was measured by a modified method of Sowbhagya and Bhattacharya (1971). About 100 mg of sample was weighed into a 100-ml volumetric flask, and 1 ml of ethanol was added to wet the sample. Then 10 ml of 0.5*N* KOH was added to the sample, and the sample was held overnight at room temperature. The sample was then diluted to 100 ml with distilled water and again held overnight at room temperature. Then, 5 ml of diluted solution was pipetted into a 100-ml volumetric flask and three drops of phenolphthalein (0.1% in 95% alcohol) were added. The starch solution was then neutralized using 1*N* HCl. Two milliliters of 0.2% iodine solution in 2% KI was added to the neutralized solution and made to volume with distilled water. The absorbance of the solution was read at 630 and 520 nm after 30 min using a DU 60 Beckman spectrophotometer. A calibration curve was prepared using mixtures of standard amylose and amylopectin solutions (McCready and Hassid 1943). Standard amylose was obtained from the National Center for Agricultural Utilization Research, Peoria, IL (C. A. Knutson, *personal communication*). Apparent amylose content was estimated by dividing the mass of true amylose, as calculated from the standard curve, by starch mass.

The starch-iodine complexes were scanned for absorbance determination >400–700 nm. The wavelength showing the maximum absorbance was λ_{max}. The ratios of absorbances at 630 and 520 nm were also reported.

Differential Scanning Calorimetry

The thermal properties of the starch-lipid extrudates were analyzed using differential scanning calorimetry (DSC). The DSC thermograms were obtained with a Perkin Elmer model 2 differential scanning calorimeter (Polymer Laboratories, Pittsburgh, PA). The instrument was calibrated with indium using an empty pan as reference.

The sample preparation included weighing 2–3 mg of ground extrudate in a tared DSC pan (0219-0062). Enough water was added with a microsyringe to obtain 20% solids in the mixture. DSC pans were hermetically sealed with a sample-encapsulating press. The samples were heated from room temperature to 160°C at a rate of 10°C/min in presence of nitrogen.

Calculated values for initial peak temperature (T_i), peak temperature (T_p), and enthalpy (ΔH) were recorded for starch gelatinization and starch-lipid complex melting endotherms.

Expansion Ratios, Bulk Density, and Water Solubility Index

The radial expansion ratios of the starch extrudates were calculated by dividing the average cross-sectional area of the extrudate by the cross-sectional area of the extruder nozzle. Each value was an average of 10 readings.

The bulk densities of the extrudates were calculated as:

$$\rho = 4/\pi d^2 l \qquad (1)$$

where ρ = bulk density (kg/m^3), d = diameter of the extrudate (m), and l = length of 1 kg of extrudate (m).

Water solubility index was determined as described by Anderson et al (1969) and was expressed as percent of dry weight of the sample.

Statistical Analyses

Data obtained in this study were analyzed using statistical software (version 6.0, SAS Institute Inc., Cary, NC). To determine whether a single model could be used to explain the behavior of starches extruded with or without stearic acid, *F*-statistics obtained by Equation 2 were compared with tabulated values.

$$F = \frac{\dfrac{SSE_{AB} - (SSE_A + SSE_B)}{DFE_{AB} - (DFE_A + DFE_B)}}{\dfrac{SSE_A + SSE_B}{DFE_A + DFE_B}} \qquad (2)$$

where SSE is sum of squares for error and DFE is degrees of freedom for error. The subscripts A and B refer to models for samples extruded with and without stearic acid, respectively, with temperature, screw speed, and moisture content and their interaction terms in the model. Subscript AB refers to a model containing the lipid term in addition to factors included in models A and B. All observations were used to fit model AB. The lipid term in the model had two levels: level 1 for samples with stearic acid, and level 2 for samples without stearic acid.

To determine the significance of different factors as a group against the remaining factors, the *F* statistic was determined and compared with the tabulated value:

$$F = \frac{\dfrac{SSE_{AB} - SSE_C}{DFE_C - DFE_{AB}}}{\dfrac{SSE_{AB}}{DFE_{AB}}} \qquad (3)$$

where subscript AB refers to the model discussed above and subscript C refers to the reduced model, excluding the factor being tested and its interaction terms with other factors.

RESULTS AND DISCUSSION

Differential Scanning Calorimetry

DSC data indicated that extrusion of normal starch with stearic acid formed a complex between the amylose portion of the starch and the stearic acid. This complex was formed at all processing conditions, as indicated by an endothermic peak at 107–112°C. When samples were cooled and reheated, these peaks reappeared in the DSC scans, confirming that these peaks were caused by the formation of the complexes. Preliminary experiments showed that enthalpy changes, as measured by DSC, did not correlate with complexed amylose as measured by iodine binding capacity. Schweizer et al (1986) also observed that endothermic transition enthalpies could not be used to quantify the amylose complex formation. Galloway et al (1989) also used DSC but did not report any quantitative values of complex formation during twin-screw extrusion of wheat flour with lipids. Hence, DSC was used only as a qualitative indicator of complex formation. In this study, iodine binding capacity and apparent amylose contents were used to quantify extent of complexing.

Extruded starches did not show any endothermic transition at 60–70°C, indicating complete gelatinization of starch. Starch extruded without added stearic acid either either did not show a peak at 107°C or showed a very small endotherm, which was attributed to the melting of a complex between native lipids and amylose.

Iodine Binding Capacity

The effects of barrel temperature, screw speed, and moisture content on iodine binding capacity of normal corn starch, extruded without and with stearic acid, are shown in Figures 1 and 2. The statistical analysis showed that only lipid (the presence or absence of stearic acid) and temperature factors were significant; all the interactions were insignificant. As the lipid factor was highly significant, the response surfaces (with and without lipid) were essentially parallel. This was further confirmed by *F*-statistics testing the parallelism of the models fitted with and without lipids (Equation 2), which indicated that the same model could be used for samples extruded with and without stearic acid.

For starches extruded without stearic acid, the iodine binding capacity of starches stayed between 3.5 and 4 mg/100 mg (Fig. 1). Upon addition of stearic acid before extrusion, the iodine binding capacity decreased to 2.5–3.5 mg/100 mg (Fig. 2). The decrease in iodine binding capacity has two possible causes: 1) the formation of amylose-stearic acid complexes, or 2) breakdown of amylose chains to such an extent that helices could not be formed. The second possibility was discarded because lipids generally act as lubricants inside the extruder; hence, we would not expect increased degradation of amylose chains in the presence of lipids. Therefore, the decrease in iodine binding capacity was attributed to formation of amylose-stearic acid complexes, as confirmed by DSC.

For starch extruded with stearic acid at a screw speed of 140 rpm, iodine binding capacity values decreased when barrel temperatures were increased to 110–140°C. However, with a further increase in barrel temperature, they increased. This trend was observed for all moisture contents (Fig. 2).

The effect of temperature can be explained from behavior of extruded starches in DSC studies. The melting temperature of complexes was ~107°C. At higher extrusion temperatures such as 170°C, the melt temperature was >170°C, which is much higher than 107°C. Therefore, the complexes could have been dissociated before leaving the die of the extruder. The barrel temperature also affected the residence time: at higher temperatures a lot of surging was observed. This also would have contributed to reduced binding due to decreased residence time for complex formation. However, Guzman et al (1992) observed no increase in lipid binding when corn meal was extruded with triglycerides and phospholipids in a twin-screw extruder at 50–60°C and 85–90°C.

The iodine binding capacity of the extruded starch depended on the amount of specific mechanical energy input to the extruder screw ($R^2 = 0.70$) as given by the relationship:

$$\mathrm{IBC} = 1.9183 + 0.8519 \times L + 0.0006 \times E - 0.0002 \times L \times E \quad (4)$$

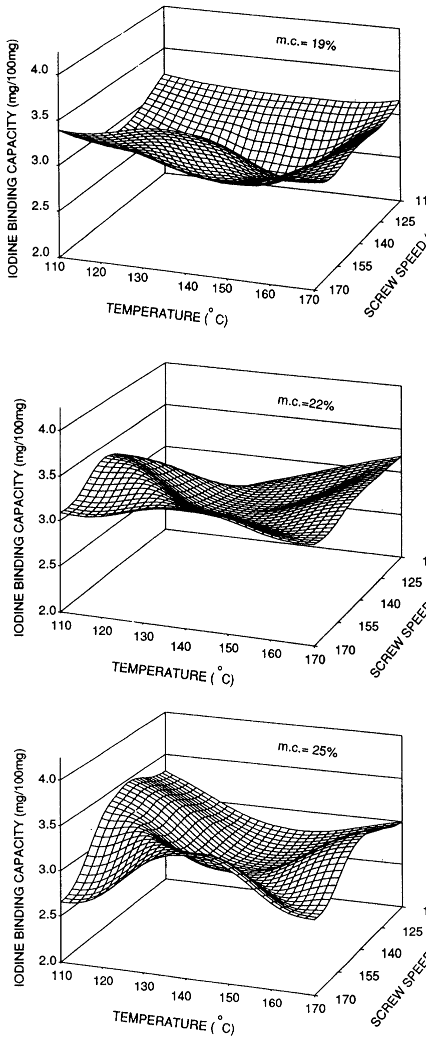

Fig. 1. Effect of temperature, screw speed, and moisture content on iodine binding capacity of starches extruded without stearic acid.

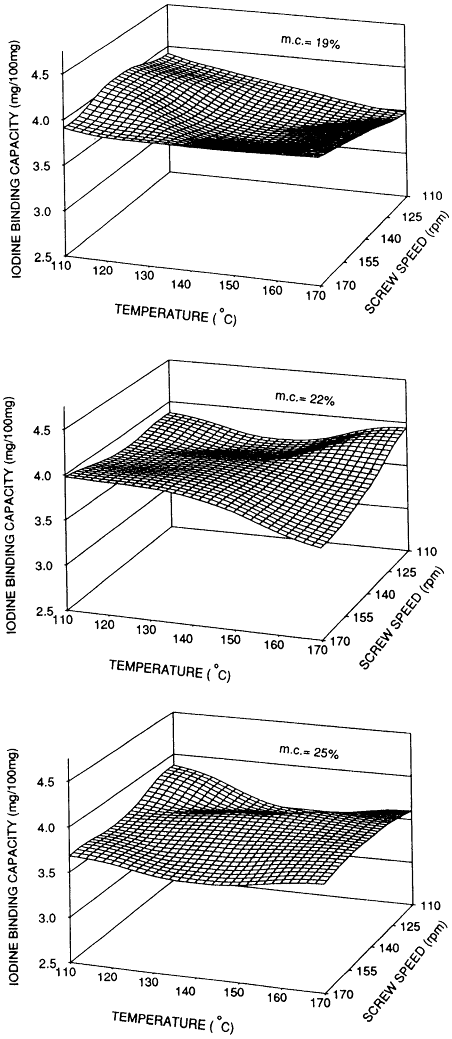

Fig. 2. Effect of temperature, screw speed, and moisture content on iodine binding capacity of starches extruded with stearic acid.

where L = lipid level (level 1 was starch with stearic acid and level 2 was starch without stearic acid); E = specific mechanical energy.

Apparent Amylose Content

Like iodine binding capacity, the apparent amylose content of the starch was affected by addition of stearic acid, decreasing 30–70% (Figs. 3 and 4). The amount of decrease was directly proportional to degree of complexing. The results of apparent amylose content confirmed those of iodine binding capacity: the same model could be used to describe the behavior of starch extruded with or without stearic acid.

For starch extruded without stearic acid, the apparent amylose content was 21–23%. There was a slight variation with the lowest temperature at 140°C. This behavior was observed at all moisture levels studied. All other factors and interactions were insignificant. When starch was extruded with stearic acid, the apparent amylose content decreased significantly for all processing conditions, with lowest values being observed for temperatures at 110–140°C. Moisture content and screw speed did not significantly affect the apparent amylose content. These results are in agreement with the observations for iodine binding capacity.

The wavelength of maximum absorption (λ_{max}) and ratio of absorbances at 630 and 520 nm were determined. For starch extruded without stearic acid, the λ_{max} was 590–599 nm. However, for starch extruded with stearic acid, the λ_{max} decreased to 540 nm. The color of the starch iodine complex changed from blue to purple or violet. The ratio of absorbances at 630 and 520 nm also decreased as the iodine binding capacity, apparent amylose content, and λ_{max} values decreased. Thus, this ratio also can be used to indicate the degree of complex formation.

The apparent amylose content was highly correlated with the specific mechanical energy input to the extruder screw (R^2 = 0.95). The regression equation was:

$$\text{Apparent amylose content} = 8.1616 + 7.2862 \times L - 0.0160 \times E + 0.0079 \times L \times E \quad (5)$$

where L = lipid level (level 1 was starch with stearic acid and level 2 was starch without stearic acid); E = specific mechanical energy.

According to observed values of iodine binding capacity and apparent amylose content, the most binding occurred at 110–140°C barrel temperature; screw speed and moisture content did not significantly affect lipid binding. The highest levels of binding for the range of experimental variables studied were at 110–140°C barrel temperature, 140 rpm screw speed, and 19% moisture content.

Effect of Lipid Binding on Properties of Extrudates

Expansion ratios, bulk density, and water solubility index properties of starch-lipid extrudates were significantly affected by the presence of stearic acid and by processing conditions. These three properties were highly correlated ($R^2 > 0.88$). Bulk density was inversely related to the expansion ratio. These properties were not correlated with the iodine binding capacity and apparent amylose content values, as observed earlier.

Figures 5–8 show the effects of stearic acid and processing conditions on expansion ratio and water solubility index of extrudates. The F-statistics (Equation 2) suggested that, unlike iodine binding capacity and apparent amylose content, two different models were required to describe the behavior of starch extruded with and without stearic acid.

All processing variables affected the expansion ratio, bulk

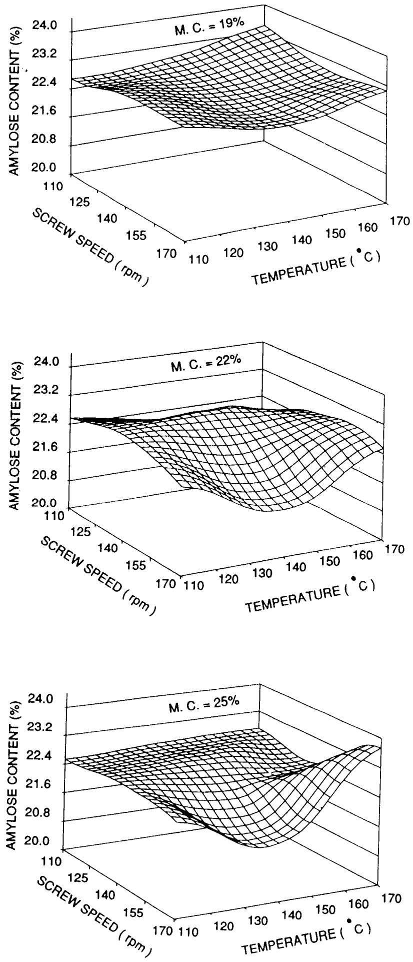

Fig. 3. Effect of temperature, screw speed, and moisture content on apparent amylose content of starches extruded without stearic acid.

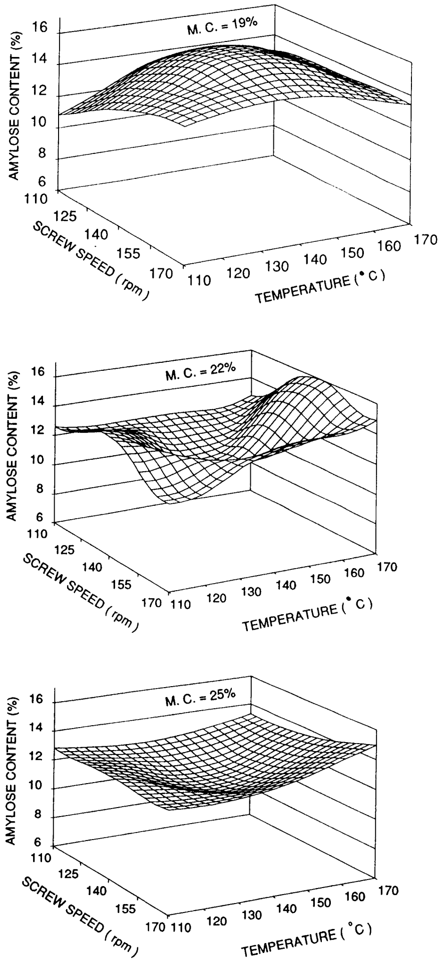

Fig. 4. Effect of temperature, screw speed, and moisture content on apparent amylose content of starches extruded with stearic acid.

density, and water solubility index of the starch extruded without stearic acid. The expansion ratio varied between 4 and 13, depending on the temperature. The temperature effect was dependent on the moisture content and screw speed because temperature × screw speed and temperature × moisture content interactions were significant. The expansion ratio was highest for 19–22% moisture content, 140°C barrel temperature, and 170 rpm screw speed. Chinnaswamy and Hanna (1988) reported similar values of expansion ratios for 25% amylose starch while optimizing processing conditions for expansion of corn starch in a single-screw extruder. Bulk densities and expansion ratios were inversely related to each other and, therefore, were affected by the same dependent variables.

Addition of stearic acid formed complexes that decreased expansion of the product, increased bulk density, and reduced water solubility index. Except for temperature, all other factors and interaction terms in the model became insignificant. The higher expansion ratio values were obtained for higher temperatures or lower screw speeds. Higher temperatures decreased the level of lipid binding and, therefore, increased expansion ratio and decreased bulk density values. The bulk density values were as high as 1,700 kg/m^3 at 110°C, and as low as 500 kg/m^3 at 170°C. This information may be useful in controlling properties of extruded products such as fish feeds where flotation needs to be controlled.

Several authors have reported that the decreases in expansion of the starches extruded without lipids were affected by moisture content (Seiler et al 1980, Faubion and Hoseney 1982, Antila et al 1983, Owusu-Ansah 1983, Guy and Horne 1988) and by barrel temperature (Alvarez-Martinez et al 1988). Bulk density increased with an increase in extrusion moisture content (Hayter et al 1987). Water solubility index increased with: 1) severity of thermal treatment in the extruder (Meuser et al 1987, Colonna et al 1989); 2) a decrease in moisture content (Anderson et al 1969, Mercier and Feillet 1975, Gomez and Aguilera 1984, Paton and Spratt 1984); and 3) an increase in specific mechanical energy input (Meuser et al 1987). The results of the present study confirmed some of those findings. However, the temperature effect was quadratic for all functional properties studied in this experiment.

Very little is known about the effect of processing conditions on expansion of starches extruded with lipids. Galloway et al (1989) reported that only barrel temperature influenced the expansion of starches upon twin-screw extrusion with glyceryl monostearate, whereas water solubility was affected only by screw speed. The present study suggests that in addition to screw speed, the water solubility index was also affected by extrusion temperature.

Table I gives prediction equations for iodine binding capacity, apparent amylose content, water solubility index, and bulk density. These equations can help in selecting processing conditions for particular end-product applications. Prediction equations for expansion ratio and water solubility index of starches extruded with stearic acid showed considerable lack of fit and low R^2 values (data not reported).

CONCLUSIONS

The results obtained in this study suggest that extrusion temperature was the most significant factor affecting amylose-lipid complex formation. Highest levels of lipid binding occurred at barrel temperatures of 110–140°C, 140 rpm screw speed, and 19% feed moisture content. A single model was used to explain changes in the iodine binding capacity and apparent amylose content of

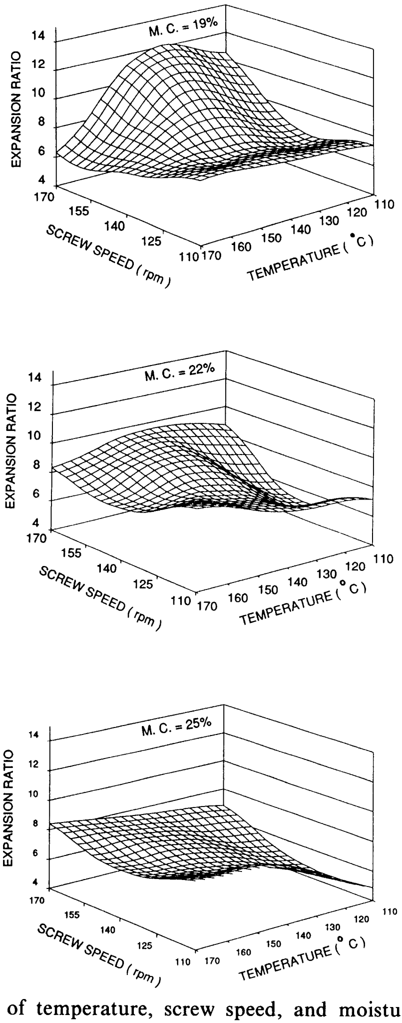

Fig. 5. Effect of temperature, screw speed, and moisture content on expansion ratio of starches extruded without stearic acid.

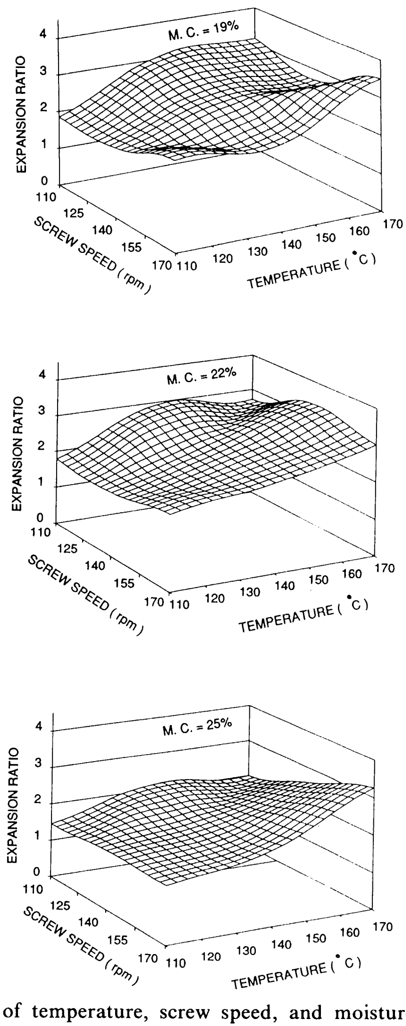

Fig. 6. Effect of temperature, screw speed, and moisture content on expansion ratio of starches extruded with stearic acid.

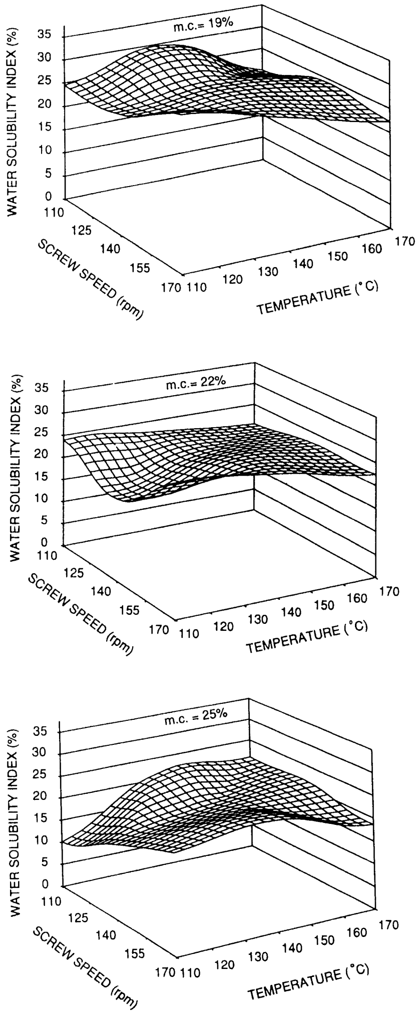

Fig. 7. Effect of temperature, screw speed, and moisture content on water solubility index of starches extruded without stearic acid.

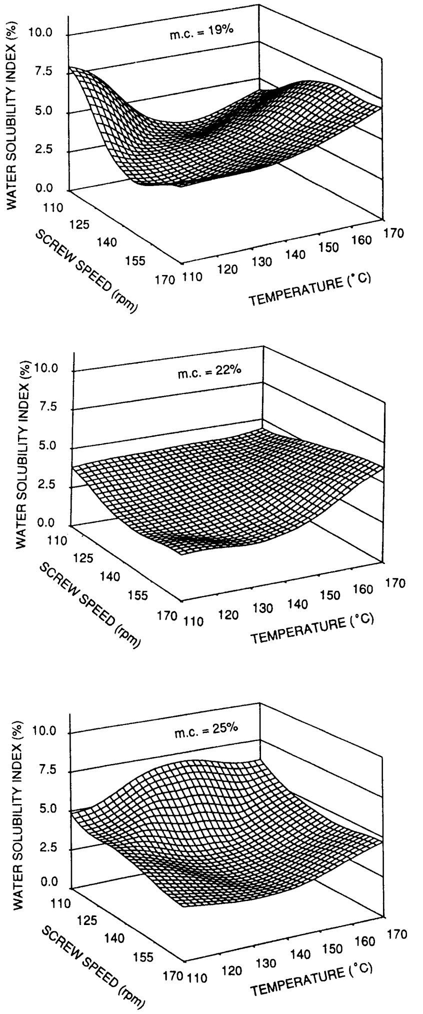

Fig. 8. Effect of temperature, screw speed, and moisture content on water solubility index of starches extruded with stearic acid.

TABLE I
Regression Equations for Specific Functional Properties

Property	Regression Equation[a]	R^2
Iodine binding capacity	$2.52988 + 0.85611L - 0.02106T + 0.017265R + 0.0058M + 0.0000518T^2 - 0.00002R^2 + 0.002222M^2 + 0.0000469TR$	0.74
Apparent amylose content	$6.74926 + 9.87815L + 0.006666T + 0.12294R - 1.30905M + 0.0002644T^2 - 0.00017696R^2 + 0.039156M^2 - 0.00016883TR - 0.0015525TM$	0.94
Expansion ratio (without stearic acid)	$14.76963 + 0.017151T - 0.033043R - 0.370123M - 0.0004512T^2 + 0.000614R^2 - 0.01577M^2 - 0.000505TR + 0.008315TM - 0.0025694RM$	0.60
Bulk density (with stearic acid)	$5.57832 - 0.0406736T - 0.01289R - 0.026831M + 0.0000693T^2 - 0.00000696R^2 + 0.0016934M^2 0.0001055TR - 0.0002688TM - 0.00001358RM$	0.76
Bulk density (without stearic acid)	$1.043167 - 0.005405T + 0.0016537R - 0.0341574M + 0.00002574T^2 - 0.000021481R^2 0.0022037M^2 + 0.00002694TR - 0.0003773TM - 0.000009259RM$	0.85
Water solubility index (without stearic acid)	$32.6726 + 0.56123T - 0.122617R - 2.7829M - 0.0030716T^2 + 0.0012821R^2 - 0.0553086M^2 - 0.001959TR + 0.0255TM + 0.004213RM$	0.79

[a] T = temperature (°C), R = screw speed (rpm), and M = moisture content (%). L = lipid level (level 1 with stearic acid and level 2 without stearic acid).

starches extruded with and without stearic acid. However, different models were required to describe expansion ratio, bulk density, and water solubility index. For starch extruded with stearic acid, these properties were mainly dependent on barrel temperature. For starch extruded without stearic acid, all the processing variables significantly affected these properties.

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