Physicochemical Changes in Cornstarch as a Function of Extrusion Variables

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ABSTRACT

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The effect of primary extrusion variables, ie, temperature, feed moisture, and screw speed, on the gelatinization, water absorption index, water solubility, and cooked viscosity of cornstarch was studied. All the physicochemical characteristics measured except water solubility were significant in their first or second order terms. The responses measured were linearly and quadratically related to the variables and accounted for more than 90% of the total variation. Water solubility was not quadratically related to the extrusion variables but increased with increasing temperature and moisture content. The overall physicochemical results indicated some hydrolytic breakdown of starch during extrusion.

Over the past 20 years, the use of food extruders has increased, mainly because of greater demand for snack and convenience foods. The introduction of twin-screw extruders widened the scope of food extrusion in the manufacture of cereals and starches, ready-to-eat cereals, infant formulas, snack foods, soft moist pet foods, breadings, and coatings. Despite the increased use of extrusion processing on starch-based products, relatively little is known about the effect of extrusion variables on the physicochemical characteristics of the product. Earlier studies (Anderson et al 1969a, 1969b, 1970, 1971; Conway 1971a, 1971b; Conway and Anderson 1973) were done with the aim of producing specific food products. Subsequent work on the twin-screw extruder (Charbonniere et al 1973, Mercier 1977, Mercier and Feillet 1975, and Mercier et al 1980) has clarified the effect of some extrusion variables on the physicochemical changes of starch. With the increased use of twin-screw extruders in the manufacture of starchy products and starch-based food ingredients, the practice of empirical modification of operating conditions, which yield neither optimum products nor lower cost, cannot be continued. The objective of this article is to define the major extrusion variables responsible for physicochemical changes in extruded cornstarch and to derive appropriate model equations relating these variables to the induced changes.

MATERIALS AND METHODS

Raw commercial cornstarch was obtained from St. Lawrence Starch Co. (Mississauga, Ont.). Analyses in this laboratory indicated that the starch contained 0.26% protein (N × 5.88), 0.4% fat, and 10.9% moisture. High performance liquid chromatography (HPLC) acetonitrile was obtained from Fisher Scientific Co. (Fairlawn, NJ) and highly purified maltose and glucose from Supelco Inc. (Bellefonte, PA).

A Creusot-Loire model BC 45 fitted with corotating, intermeshing twin screws and a barrel heated by an electrical induction system was used in this study. The screw profile consisted of long feeding screws, short feeding screws, one (for low pressures) or two (for high pressures) pressure building screws, and mixing screws. The water delivery system, the feed screw on the extruder, and the feed-hopper screw, respectively, were calibrated individually, and a computer program was written to determine the settings required to deliver raw material of a desired moisture content. A 5-min running time was allowed for equilibration before samples were taken. Samples were collected, air-dried, milled on a laboratory hammer mill, cooled with ice to pass a 200-mesh screen, and stored in sealed plastic bags until needed for analysis.

The extent of gelatinization of the extrudate was determined by the method of Birch and Priestly (1973), with modifications described by Owusu-Ansah et al (1982a). Gelatinization is reported as the absorbance ratio of the amylose-iodine complex for samples dispersed in 0.25*M* KOH compared to respective samples dispersed in 0.7*M* KOH. Water absorption index (WAI) was determined in triplicate by the procedure described by Anderson et al (1969a) and is reported as grams of gel per gram of dry extrudate. Viscosity was measured on a Brabender viscoamylograph under the conditions suggested by Anderson et al (1969a) and with heating conditions described by Mercier and Feillet (1975). Both the initial (at 25°C) and final (at 50°C) cooked paste viscosities were determined from the amylograph curves and are given in Brabender units (BU).

Ten-milliliter aliquots of fresh, distilled water were used to extract water-soluble carbohydrates four times from 1.0 g of the dried milled extrudate. These extracts were combined, concentrated in a rotary film evaporator, and examined by paper chromatography using *n*-propanol-ethanol-water (7:2:1, v/v) (Takaya et al 1979) in the presence of alkaline acetone-silver nitrate (Trevelyan et al 1950) for detection. Water-soluble carbohydrates were hydrolyzed to glucose according to the procedure of Chiang and Johnson (1977a). The hydrolysate was passed through a mixed-bed ion-exchange resin and glucose determined by HPLC (Water Associates, Inc., Milford, MA). Acetonitrile-water (80:20) was used as the HPLC solvent at a flow rate of 2.0 ml/min and the carbohydrate detected using a refractive index detector. Total glucose in the hydrolysate was equated to the percentage of soluble solids in the milled extrudate. The water solubles in the extrudate were also compared qualitatively by sedimentation velocity ultracentrifugation. Scanning electron microscopy and the procedure described by Owusu-Ansah et al (1982a) were used in examining the milled extrudates.

A central composite rotatable response surface design (Mullen and Ennis 1979) was used, with the overall ranges and selected variables tabulated in Table I. The general linear models (GLM) option of the SAS program (SAS Institute, Inc., Raleigh, NC) were used to analyze data, and the SCIP04 (University of Guelph, Ont.) three-dimensional plotting program was used to plot the generated regression equations.

RESULTS AND DISCUSSION

The effect of extrusion variables, temperature (°C), feed moisture (%), and screw speed (rpm) on the physicochemical properties of the extruded corn starch are presented in Table II. Because the design could be rotated, the first and second orders and

TABLE I
Operating Ranges of Extruder Process

Variables	Extruder Conditions				
Temperature, °C	100	128	170	212	240
Moisture, %	11	13.4	17	20.6	23
Screw speed, rpm	50	58	70	82	90

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the interactions of each variable are independent of each other. The contribution of each factor can be evaluated by its contribution to R^2 (Table II), the higher R^2 values indicating the relatively greater importance of the response measured. The corresponding regression equations are given in Table III, and the visual three-dimensional representations of selected responses of the physicochemical parameters to the extruder variables are given in Figs. 1 and 2.

The selected variables proved to be those related to starch gelatinization, with the model regression equation accounting for more than 90% of the variation in this physicochemical parameter (Table II). Analysis by stepwise regression indicated that the cognizant factors were the temperature-moisture interaction, temperature, moisture, and screw speed. The response-surface diagrams generated from the regression equations at screw speeds of 50, 70, and 90 rpm (Fig. 1a, 1–3), showed that at 90 rpm maximum, gelatinization (1.2 absorbance ratio) occurred at lowest temperature (100°C) and highest feed moisture (23%), whereas minimum gelatinization (0.13 absorbance ratio) was observed at the same temperature but at the lowest feed moisture (11%). The minimum cannot be seen at the two lower screw speeds (Fig. 1a, 1 and 2) because they occur at the point of high moisture and temperature, which cannot be seen in the figure. These results are anomalous because gelatinization would be expected to increase at higher levels of temperature and moisture. Lawton et al (1972) observed similar trends in extruded cornstarch and attributed this effect to the lubrication provided by excess water. This would probably not explain the present case, considering the improved mixing capability and deeper flights of the screws in the twin-screw extruder. It seems more likely that screw profile was a factor in this anomalous result. The "high pressure building screw profile" with a greater compression ratio probably generated greater mechanical work on the starch such that complete gelatinization (alkali solubilization) could be attained even at the minimum temperature of 100°C. Any further increase in temperature did not affect gelatinization per se, but rather increased the solubility of the product. Since the measurement of gelatinization was based on amylose-iodine complex formation, the production of lower molecular weight carbohydrate would result in a corresponding decrease in the absorbance ratio and thus reduce the apparent gelatinization.

To ascertain whether complete gelatinization in all samples had occurred, the samples were subjected to SEM examination. Micrographs (Fig. 3) showed the absence of intact or partially disintegrated starch granules even at 100°C. Solubility studies showed that water solubility increased with increased temperature and moisture content (Fig. 4) and that although a reduction in screw speed increased water solubility at higher moisture contents, this had little or no effect at lower moisture contents. The water-soluble fraction analyzed by paper chromatography indicated the presence of maltose and other low molecular weight carbohydrates in the samples processed at 170°C, 23% feed moisture content, and a screw speed of 70 rpm. An examination of the water-soluble fraction of the extrudates by sedimentation velocity ultracentrifugation showed a slowly sedimenting and rapidly diffusing peak for the anomalous high-moisture, high-temperature sample, indicating the presence of a substantial amount of low molecular weight material. This sample was far more readily attacked by α-amylase (Owusu-Ansah et al 1982b), apparently because it was partly hydrolyzed. These observations implied that although partial hydrolysis might not predominate in extruded cereal starches, specific processing conditions may yield such end products.

Some hydrolysis might occur during extrusion of cereal starches or grits (Chiang and Johnson 1977b). Maltose, glucose, and arabinose have also been observed in extruded cornstarch (Henderson, *unpublished data*). Mercier and Feillet (1975), however, discounted the presence of such hydrolytic products in extruded cereal starches. Present results partially confirm previous observations, and it may be possible that different processing conditions, eg, screw profile, might account for these discrepancies.

To determine whether the screw profile used could be accountable for the anomalous gelatinization, another screw profile was used. When a "low-pressure profile," a fixed moisture content (17.0%), and a screw speed of 70 rpm were used, maximum gelatinization occurred at about 170°C (Fig. 5). Because of the relatively low pressures and the lower mechanical work associated with this second profile, a higher temperature was needed for complete gelatinization. The screw profile was shown to be an important variable and may have been partly responsible for the observed anomaly in the gelatinization studies.

The effect of the extrusion variables on WAI is shown in Table II. As with the gelatinization results, the interaction between temperature and moisture was the most significant variable. When this is removed, temperature becomes the next significant variable ($P \leq 0.01$), followed by moisture and then screw speed. The second order terms of moisture and temperature, and the interaction between temperature and screw speed were also significant at the 5% level. The model equation predicting this response (Table III) accounted for 92.83% of the total variation ($P \leq 0.01$). The response diagrams generated from this equation (Fig. 1b) showed a similar trend to those observed for gelatinization. Maximum WAI was found at 100°C and 23% moisture content at 90 rpm.

TABLE II
Contribution and Significance of Extruder Variables to the Physicochemical Properties of Extruded Cornstarch

Independent Variables	Percent Contribution to R^2: Gelatinization	Water Absorption Index	Cold Viscosity	Cooked Viscosity
Temperature × moisture	28.08[a]	27.69[a]	7.98[a]	8.29[a]
Temperature	25.52[a]	24.59[a]	13.23[a]	19.73[a]
Moisture	14.20[a]	14.04[a]	61.89[a]	40.84[a]
Screw speed	12.02[a]	11.60[a]	1.04	6.22[b]
Temperature × screw speed	3.91	3.78[b]	0.13	0.48
Moisture × moisture	3.89	6.68[b]	0.12	6.24[b]
Temperature × temperature	2.11	4.02[b]	8.01[a]	10.36[a]
Screw speed × screw speed	1.71	0.39	0.05	0.18
Screw speed × moisture	0.04	0.04	0.01	0.17
R^2 for regression	91.47[a]	92.83[a]	92.46[a]	92.51[a]

[a] $P \leq 0.01$.
[b] $P \leq 0.05$.

TABLE III
Regression Equations for the Various Responses

Response	Equation[a]
Gelatinization	$Yg = -22.268 + 1.062X_1 + 24.099X_2 - 4.598X_3 - 0.022X_1^2 - 0.257X_2^2 + 0.016X_3^2 - 0.084X_1X_2 + 0.009X_1X_3 + 0.011X_2X_3$
Water absorption index	$Ya = -4.392 + 0.052X_1 + 1.096X_2 - 0.136X_3 - 8.865 \times 10^{-5} X_1^2 - 1.415 \times 10^{-2} X_2^2 + 3.140 \times 10^{-4} X_3^2 - 3.356 \times 10^{-3} X_1 X_2 + 3.720 \times 10^{-4} X_1 X_3 + 4.670 \times 10^{-4} X_2 X_3$
Cold viscosity	$Yv_1 = -1839.088 + 12.378X_1 + 94.422X_2 + 3.436X_3 - 0.021 X_1^2 - 0.373 X_2^2 - 0.021 X_3^2 - 0.322 X_1 X_2 - 0.012 X_1 X_3 + 0.029 X_2 X_3$
Cooked viscosity	$Yv_2 = -1022.029 + 7.541X_1 + 80.080X_2 - 3.688X_3 - 0.013X_1^2 - 1.300 X_2^2 + 0.020X_3^2 - 0.174X_1 X_2 - 0.012X_1 X_3 + 0.087 X_2 X_3$

[a] Where X_1 = temperature, X_2 = moisture, and X_3 = screw speed.

Intermediate temperatures and moisture contents and the extremes of these two variables showed a minimum WAI at 50 and 70 rpm. Screw profile also had an effect on WAI with the low pressure profile, producing a maximum WAI around 170°C, similar to gelatinization (Fig. 2).

The overall effects of the extrusion variables on cold and cooked viscosities are also presented in Table II. For cold viscosity, feed moisture content was the most significant variable. When this is removed, temperature then becomes the next significant variable ($P \leqslant 0.01$) followed by the second order of temperature and then the interaction between temperature and feed moisture content. The predicting model regression equation (Table III) accounted for

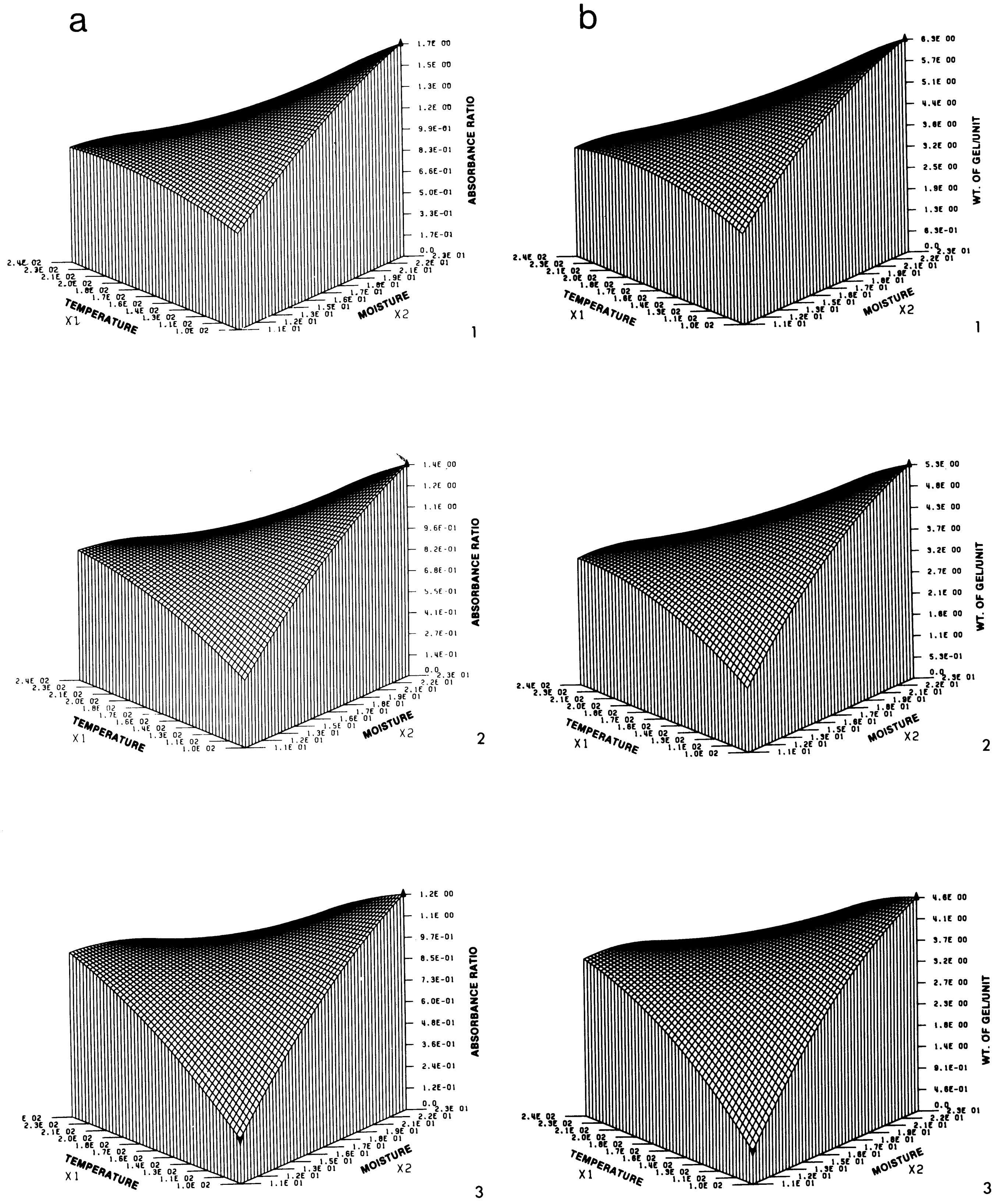

Fig. 1. Response-surface diagrams illustrating the effect of temperature, moisture, and screw speed on **(a)** gelatinization and **(b)** water absorption index for responses generated at **(1)** 50, **(2)** 70, and **(3)** 90 rpm.

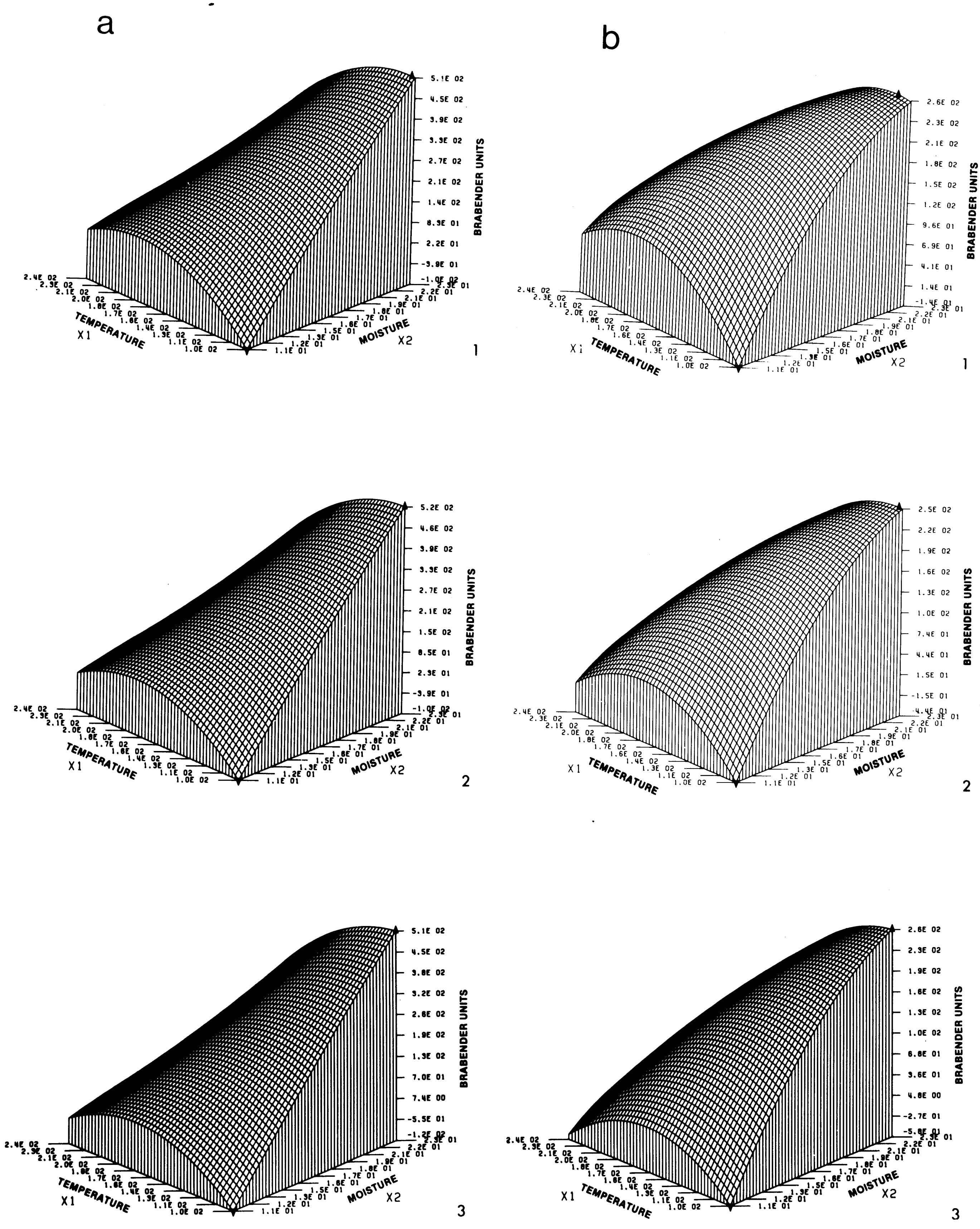

Fig. 2. Response-surface diagrams illustrating the effect of extrusion variables on **(a)** cold and **(b)** cooked viscosities for responses generated at **(1)** 50, **(2)** 70, and **(3)** 90 rpm.

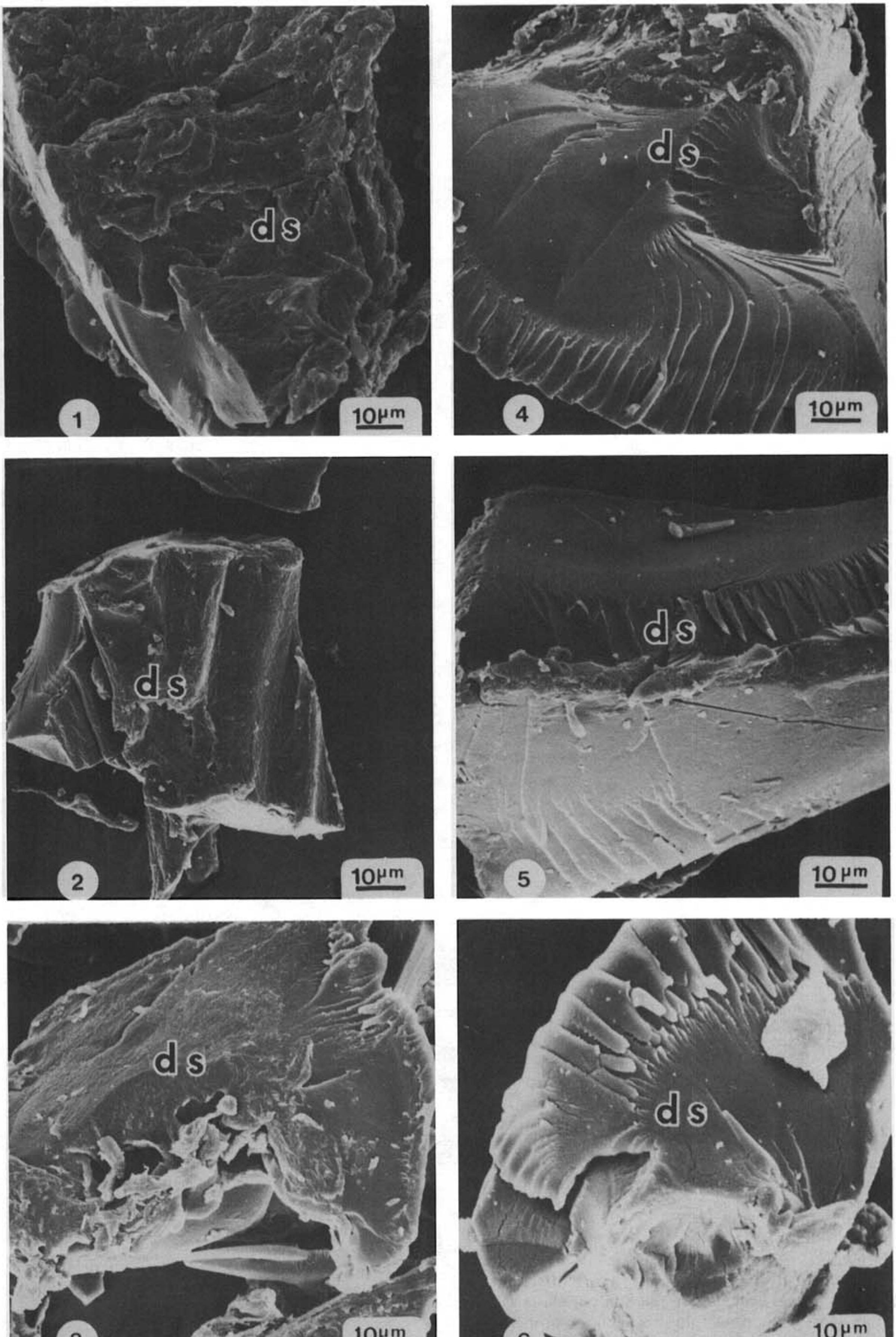

Fig. 3. Scanning electron micrographs of milled extruded corn starch showing disintegrated starch **(ds)**. Coded numbers in the micrographs indicate the extrusion parameters, temperature (° C), moisture (%), and screw speed (rpm) used. **1,** 10, 17.0, 70; **2,** 240, 17.0, 70; **3,** 170, 23.0, 70; **4,** 170, 17.0, 70; **5,** 212, 20.6, 58; **6,** 128, 13.4, 58.

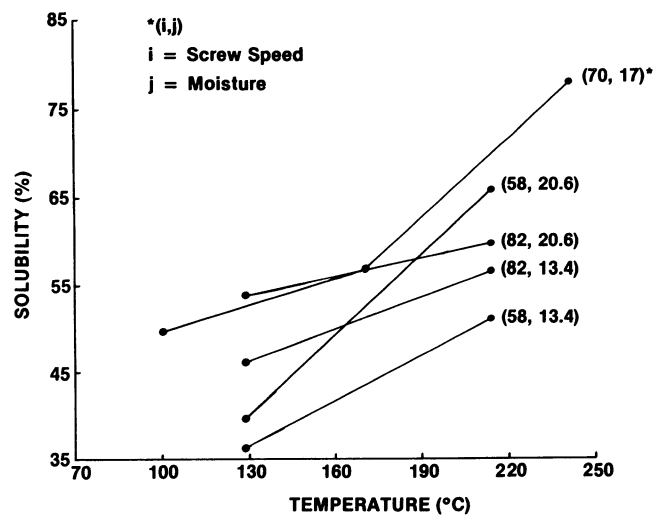

Fig. 4. Changes in the water solubility of extrudates as a function of extrusion temperature, moisture, and screw speed.

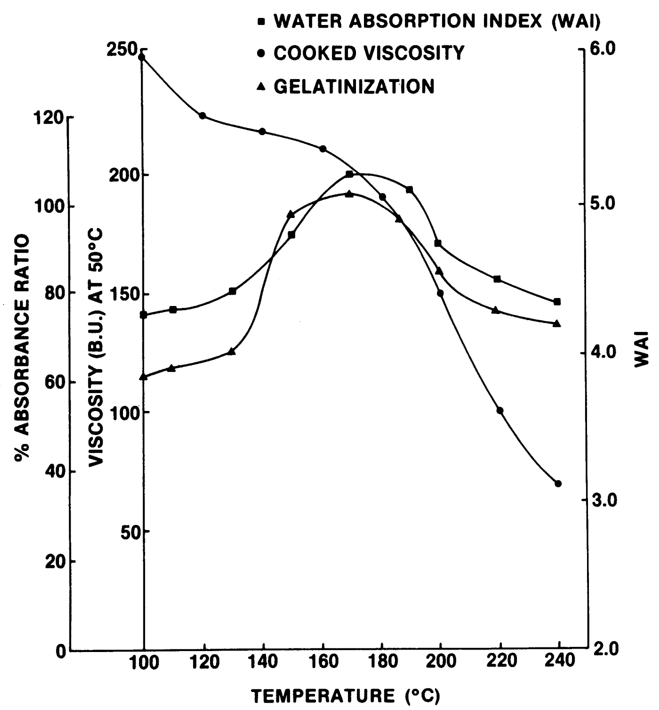

Fig. 5. Changes in gelatinization, water absorption index, and cooked viscosity as a function of temperature, using a low-pressure screw profile, 17% moisture, and 70 rpm screw speed.

92.46% of the total variation and was significant at $P = 0.01$. For cooked viscosity, the type and sequence of the significant variables were the same as for the cold viscosity, except that two other variables—screw speed and the second order of the feed moisture content—were also found to be significant at $P = 0.05$. The model equation predicting this response accounted for 92.15% of the total variation.

CONCLUSION

The three major extrusion variables—temperature, feed moisture, and screw speed—were significant factors affecting the physicochemical changes evaluated in this study. Gelatinization, generally the most important physicochemical property of starches, appeared to be affected in an anomalous manner, decreasing with increasing temperature. The choice of screw profile was shown to be the cause, leading to complete gelatinization at low temperature, probably because of mechanical shear associated with the profile used. Under these conditions, further heating led to the formation of water-soluble carbohydrate not accounted for in the Birch and Priestley method of determining the degree of gelatinization. When a low-pressure profile was used, the expected increase in gelatinization with increasing temperature was observed. This study has demonstrated that by using the central composite rotatable design, predictive relationships can be determined for specific physicochemical characteristics. Based on the interrelationships of desirable physicochemical characteristics relative to the major extruder variables, the processor can then use prediction equations to choose conditions that will yield an optimal product.

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