CHARACTERIZATION OF EXTRUDED WHEAT FLOUR

by

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B. S., Kansas State University, 1971

A MASTER'S THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Grain Science and Industry

KANSAS STATE UNIVERSITY
Manhattan, Kansas

1973

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INTRODUCTION

There are many methods of cooking cereal grains, some of them as old as fire itself. Most cooking processes result in the gelatinization (swelling) of the starch granules present. During cooking, there is a marked increase in digestibility and an improvement in palatability. In recent years, extrusion cooking has become one of the most important new processes developed in the food and feed industries.

This investigation was undertaken to determine some of the physical characteristics of the extrudate and to learn something about starch gelatinization caused by this method of cooking.

LITERATURE REVIEW

The amount and type of cooking is very important when considering food and feed products. A properly cooked cereal, or any material used as a feed or food, is usually preferred over an uncooked product. Smith (21) indicates some of the advantages of cooking: 1) The cereal portion will be gelatinized to improve palatability and to give maximum digestibility and availability of the caloric content. 2) The thermal inactivation of growth inhibitors found in certain oilseeds and pulses will help improve palatability. 3) The cooking process will reduce microbial count and provide a more sanitary product. 4) Proper cooking will result in bonding of proteins and vitamins, that are not heat labile, along with some minerals, to the carbohydrates present. 5) Cooking of cereal based products will extend the shelf life. 6) Cooking provides an opportunity to shape, texturize and add flavorings that will improve product acceptability. These advantages may be accomplished by nearly all of the known cooking methods, either alone or with additional processing. Extrusion cooking, however, is one method where all of these advantages may be achieved without additional equipment or processing.

Extrusion cooking first became important in the food industry in the mid-1930's in the extrusion of pasta products (7). Cereal chemists found that many cereal and cereal-based products behaved similarly to plastics in the extrusion process and could be extruded in much the same manner. From this beginning of cold-forming extrusion process, the short time-high temperature cooker that is much used in the food and feed industries today evolved.

There are several advantages of extrusion cooking over some of the other methods: 1) Large volume production may be achieved on a continuous basis at a relatively low cost; 2) The short time-high temperature process seems to be less destructive to biologically useful proteins because of the very brief period of time required to reach the peak temperature (21,10); 3) The high temperature reached during the process may destroy compounds that can cause off-flavors in some of the strong tasting bean meals (e.g., soybean meals); 4) Many of the microorganisms that cause food spoilage are destroyed, even in cases where the raw materials are highly contaminated. This is due in part to the temperature of the system as well as the pressure and the shearing action caused by the screw; 5) There are some enzyme inactivations that occur, including some of those that are detrimental to the keeping qualities (21,10); 6) Extrusion cooking is a flexible system in that a variety of conditions may result in the desired product.

There are many parameters of extrusion and extruded products to be measured as can be seen in the literature. There is a relationship of texture to density, moisture and expansion which can be observed. Several methods for the study of gelatinization can be used including microscopy, amylographs and susceptibility to enzyme attack. More recently the scanning electron microscope has been used. It is impossible to study just one parameter due to the complex nature of cereal grains and their products and the interaction of parameters of the cooker-extruder.

Expansion

In examining a commercial product that is to be consumed without further processing, one of the most important factors is expansion. This

gives some indication of tenderness of the product. The ability of a cereal based material to expand when extruded is dependent upon starch content, moisture present, temperature, and pressure differential between inside and outside of the die plate (24).

Starch is the component that contributes most to expansion. This portion of cereal grain is able to expand many times its original size. It is well known that the starch granule, when heated in water, will expand as water is absorbed. It is during this swelling process that the characteristic of birefringence, the first indication of gelatinization, is lost (11). The crystalline structure of the starch granule is disrupted by absorption of water. As the temperature of the starch-water solution is increased, the granule will continue to swell and leaching occurs. In a completely disrupted system, gelatinized starch becomes a mass many times its original size (23). Gelatinization can be accomplished during extrusion process, which was the first commercial use of the short time-high temperature cooker-extruder.

The amount of expansion is directly dependent upon amount of starch present. Williams and Baer (23) found that a pure starch sample may be expected to expand about five times the diameter of the die when extruded at optimal temperature and moisture conditions. A sample that contained 65 to 75% starch could be expected to expand four times the diameter, given optimal conditions. Similarly, a material that contains only 40 to 50% starch may only expand two to three diameters and would be more subject to collapse as the steam flashes off than samples with a greater percentage of starch (23).

While the starch content is probably the most important single factor involved in expansion, moisture content is perhaps the second most important. There are two types of expansion, in relation to the amount of water present, described by Williams and Baer (23). These are wet and dry, so named for the relative amount of water present in the system. Wet expansion is a moisture range from 20 to 30%. This level can result in complete gelatinization of starch and, if cooked a long time, will yield a high viscosity gel. Dry expansion is a lower moisture content, normally a range of from 12 to 20%. Starchy material in the dry system is subjected to more thermal degradation due to the reduced amount of moisture in the system that can absorb heat.

Starch may be completely gelatinized with very little dextrin formation when moisture content is between 25 and 35% and the temperature at or near 150° C. As the temperature is reduced, so is the percent of starch that is gelatinized. On the other hand, hot dry conditions cause more dextranization to occur (23). This is more prevalent when temperatures are in the neighborhood of 200° C and moisture is 10 to 15%. Under these conditions, starch will dextranize to form up to 30% dextrin content in an expanded product. It has been reported by Murphy (9) that decomposition of carbohydrates (cellulose) may be initiated at about 100° C. This type of degradation could also be expected to occur during low moisture-high temperature extrusion.

It has been observed by Williams and Baer (24) that high moisture (25 to 35%) materials do not expand as much as those of lower moisture (10 to 15%) content. Each water droplet will form a nucleus from which, when heated, will form a chamber or cell in the product. It is thought that much of the moisture flashes off as steam when the product emerges from the die and pressure is

released. At lower moisture levels, water expands as steam to gelatinize and puff up the starch structure to many times its original size. Higher moisture material retains enough liquid as it emerges from the die to rupture the cell structure and it will not expand as does the non-ruptured cell structure (23). There is enough moisture at higher levels to allow some contraction to occur when extruded.

Another parameter to consider about expansion is the differential between the internal and external pressure. Most products are extruded 24.6 to 35.1 kg/cm^2 (350-500psi) (15). This pressure is regulated by the size of the die opening and in some cases the steam pressure inside the barrel. Sudden release of pressure allows the product to expand. As this differential decreases there is a decrease in the amount of expansion and the reverse is also true until maximum levels are reached.

Heat applied to the system may be from two sources: applied and frictional. Applied heat will usually be either steam or electric. Frictional heat is developed inside the barrel and is related to internal construction. The barrel of many extruders has grooves machined on the internal surface, spiral or parallel to the longitudinal axis, to aid in the mixing action. These grooves also increase the amount of frictional heat developed; straight grooves cause more heat than spiral grooves (18).

Throughout the years there have been many investigations using an amylograph to measure gelatinization of starches. Recently, the addition of carboxymethyl cellulose (C.M.C.) has aided in interpretation of the amylograms (22). De Muelenaere et al. (8) indicated that gelatinization is proportional to the amount of cooking which in turn can be related to the viscosity generated by the material in solution at 25° C.

The initial flat portion of the amylograph curve represents that period where swelling is insufficient to register an increased viscosity (3). This is true for native starches, but when they have been partially or fully gelatinized there is a marked difference in their behavior. Anker (3) indicates that granule swelling becomes more pronounced as the temperature is raised and causes the mixture to become more viscous. As granules continue to swell and rupture, the solution will become more viscous as the temperature increases. Once peak viscosity has been reached, continued mixing and heating result in a decreased viscosity. Important data points from an amylogram are temperature of transion (the temperature at which the first perceptible increase in viscosity occurs), maximum viscosity and temperature at maximum viscosity (5).

Conway (5), using a Brabender visco-amylograph, determined that a product that had been extruded at 190° C, 25% moisture, and a 3:1 compression screw, resulted in a completely cooked product that would form the base for a good gruel. The same material extruded under the same conditions, only at 14% moisture, exhibited a minimum cold paste viscosity for a completely cooked product and would be suitable for a beverage. Anderson et al. (2) noted that partially cooked products have low cold paste viscosity.

In a study by Anderson et al. (1), corn grits were extruded under a variety of conditions and the effects on the amylograph of final cooked paste viscosity and water solubility were examined. This study revealed that: 1) A high compression ratio gave a lower viscosity; 2) Increased temperature resulted in decreased viscosity; 3) A 15% moisture gave lower viscosity than a 20% moisture; 4) Water solubility increased as the temperature increased and a similar increase in solubles occurred as the moisture level of the raw material was lowered.

Microscopy has long been used to observe components of cereal grains. Pyler (12) presented micrographs of starch granules before, during and after gelatinization to give an indication of the size of the granule during the gelatinization process. Under a plane polarized light source, a native starch granule will exhibit a Maltese cross, a phenomenon related to crystalline structure of the granule. Lack of the Maltese cross is an indication that the granule has been altered. Reeve and Walker (13) also used microscopy to study starches of popped cereals. They noted the nearly complete disruption of the starch granules.

In recent years, a new tool has been used to obtain information about cereals and their products. The scanning electron microscope is capable of giving a three-dimensional picture of a microscopic surface.

Methodology of Extrusion

There are three general classifications for cooker-extruders used in the food and feed industries today: cold forming, low pressure cooking and shaping, and short time-high temperature extrusion. The cold forming process is a mixing and shaping process and the product usually requires additional processing. Low pressure cooking and shaping characteristically has final product temperature at the die of the extruder reduced to less than 100° C. The third system, short time-high temperature (S.T.-H.T.), completely cooks the raw material at temperatures ranging from 100° C to as high as 225° C depending upon ultimate use of the product.

The main components of a high pressure cooker-extruder are the feeder compression screw, barrel, die and heating system (see Fig. 1).

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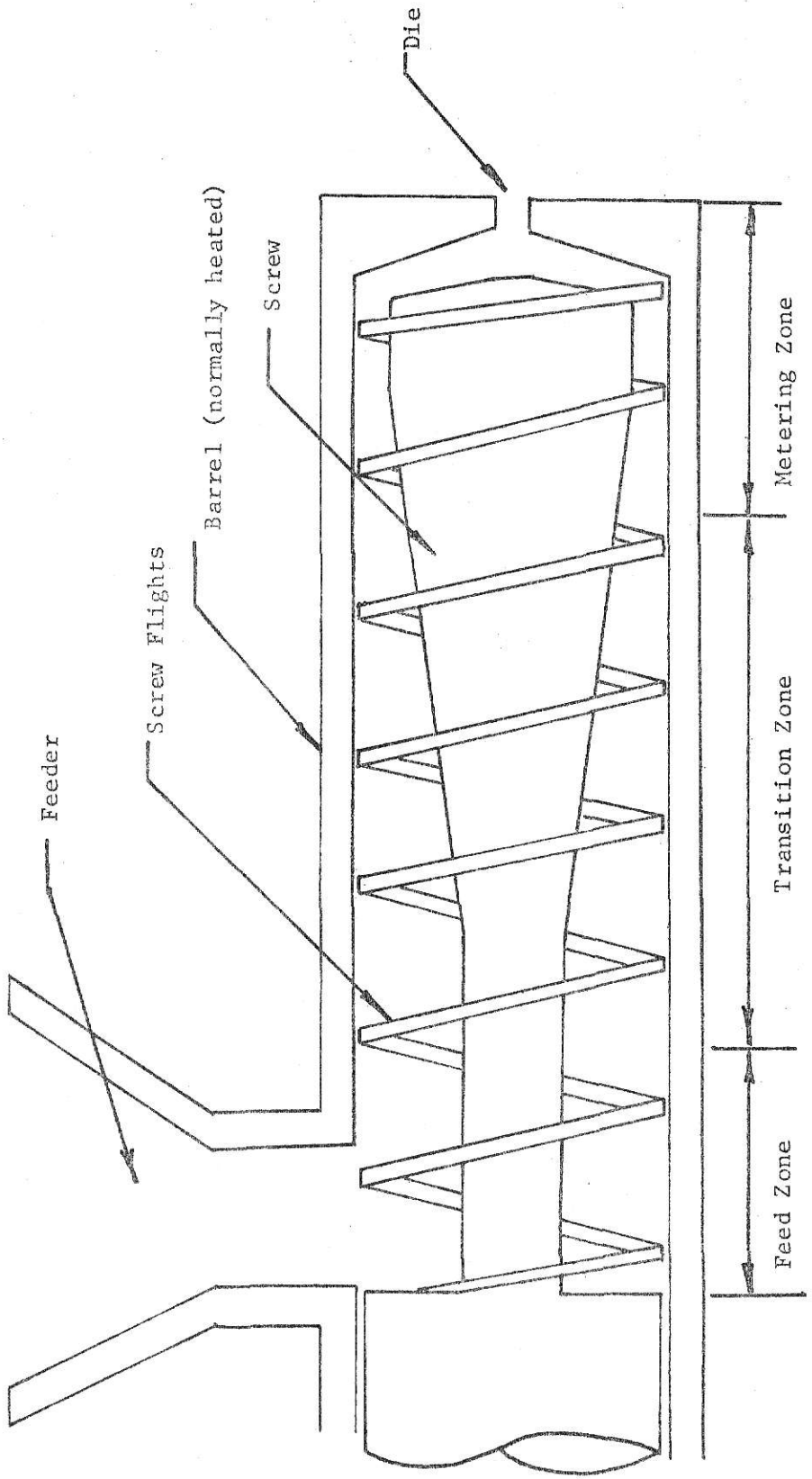

Fig. 1. The shape of a typical cooking extruder with functional sections labeled.

The raw material is presented to the extruder by the feeder which can be either of the forced or gravity flow type. The material enters the mixing section, usually the first third, where moisture can be added. In this section, the material is thoroughly mixed and presented to the second or transition section of the extruder. Here the temperature is increased and compression is initiated. At the end of this zone, the material is altered from a granular to a thermoplastic viscous material. Solid particles may exist alongside materials that have been fluxed (6). The final zone is the metering zone. In this portion of the extruder the material is completely cooked and compressed and is further mechanically worked to a fluid, semi-plastic material. From this zone the material is forced through the die and is processed as necessary (e.g., flavorings or toasting).

METHODS AND MATERIALS

The objective of this study was to evaluate some of the physical characteristics and gelatinization of starch of wheat flour extruded at a variety of moisture levels, temperatures and shear rates. Analyses were made on the outflow of the product from the extruder and notes were made on the power required to extrude at these different parameters. Examinations of samples included moisture, expansion, density, increased percent solubles, color and amylograph viscosity. Selected samples were viewed with the light microscope, the plane polarized light microscope and the scanning electron microscope. Paper chromatographs of the carbohydrate fraction were made on other selected samples.

The raw material used in this study is flour milled from a Hard Red Winter wheat at the Kansas State University Department of Grain Science and Industry pilot mill. The identification and proximate analysis of this flour is given in Table I.

The extruding equipment used in this study is a laboratory extruder manufactured by the C. W. Brabender Co. of Hackensack, New Jersey. A complete description of this instrument and its modifications is given in Table II.

Sampling

The variables chosen for extrusion study were four raw material moisture levels: 15.5%, 16.5%, 18.0%, 20.0%; four final product temperatures: 125° C, 150° C, 175° C, 200° C; and three shear rates: 60 rpm, 100 rpm, 140 rpm. The lowest and highest moisture levels were originally

Table I. Flour Proximate Analysis

KSU Flour	Ash	Protein	Moisture
72-427	6.34%	10.5%	14%

Table II. Extruder Description and Modifications

Extruder Description	
Brabender Horizontal Laboratory Extruder Model 2503	
Barrel Length	18.75 in.
Barrel Diameter	0.75 in.
Length/Diameter Ratio	25:1
Heating	one 1000 watt heater per zone
Cooling	air
Internal Surface	44.3 in^2
Barrel Volume (with 5:1 screw fitted)	47.10 in^3

Modifications

1. The extruder was fitted with thermocouples at the end of each heating zone to measure the product temperature inside the barrel.
2. The inside surface of the barrel was modified by the addition of six triangular grooves 1/8 in. deep, parallel to the longitudinal axis.

planned to be 15.0% and 19.5%, respectively, to have a constant 1.5% moisture separation. Due to equipment limitations, each of these two moisture levels were adjusted upward 0.5%.

This sampling scheme provided for a total of forty-eight samples. It was not possible to obtain all desired samples due to the extremely high torque required at low moisture and low shear rate combinations. Samples not obtained were 15.5% moisture, 60 rpm and three temperatures: 125° C, 150° C and 200° C.

Operating Procedure

The operating procedure for the Brabender Extruder was altered from the original instructions by Dr. P. A. Seib and Mark Stearns.[1] The procedure used is described below.

> The starting of the extruder was the most critical stage of the extrusion process. The extruder was adjusted so that zone 1 was cooling, zone 2 was heated to 95° C and zone 3 was heated to 125° C. These conditions were maintained for fifteen minutes or until the die cap nozzle was hot. When the die and die cap nozzle were thoroughly heated, flour of 25% moisture was started through the extruder. Once this material was flowing evenly through the extruder and an operating equilibrium had been achieved, flour of the desired moisture level was fed into the hopper. Upon completely changing the flour to the new moisture level, the temperature in zone 3 was adjusted to a level that

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[1]Professor and graduate student, respectively, Kansas State University Department of Grain Science and Industry.

would result in the desired final product temperature and the shear rate was adjusted to the predetermined rpm for the sample. At this time, the feeder was adjusted to provide optimal flow through the extruder at the lowest power requirements. Once the extruder had stabilized and flow was constant, a sample was taken and the following data recorded:

a. zone 1 temperature setting
b. zone 2 temperature setting
c. zone 3 temperature setting
d. shear rate
e. feed rate setting
f. pressure, end of zone 3 (when available)
g. product temperature, end of zone 1
h. product temperature, end of zone 2
i. product temperature, end of zone 3
j. grams of product per minute
k. voltage
l. amperes

Change to new parameters was accomplished by switching to the new operating conditions, allowing the extruder to stabilize, and collecting the sample as described above.

When each sample had been collected, approximately 150 grams were placed in polyethylene bags and stored for physical examinations. The remainder of the sample was ground on a Wiley laboratory hammer mill to pass through a 0.020 in. screen. It was from this ground portion that samples for those tests requiring a ground sample were taken.

Moisture determinations were made according to A.O.A.C. method 14.004 (4). This technique requires the sample to be dried for one hour in an air oven at 130° C. These analyses were made in the Analytical Laboratory, Department of Grain Science and Industry. Moisture content was used not only as a parameter of this investigation, but it was also necessary when calculating the dry matter in analyses for density, solubility and output of the extruder, and for the amylograph. By using the dry weight basis, more reliable values are obtained.

Expansion

Expansion was another characteristic of interest. Rather than take the total diameter of the product, expansion greater than the die opening of 1/8 in. (3.17 mm) was measured. From each sample ten random samples were selected and these were measured for diameter. The mean of these observations was used as the value for diameter in the statistical analysis.

Shear Resistance

Shear resistance is a measure of resistance a sample has to breaking. This is related to characteristics of toughness and rigidity. Many samples were soft and readily breakable. To measure shear resistance, a Brabender Extensograph was used. Random samples were selected and the force required to break the sample across a two-inch opening was recorded. This instrument measures in Brabender Units (gm-cm) or resistance and gives only a relative value for breaking strength.

Solubles

The amount of solubles in a cereal product that has been cooked is an indication of the degree of the starch gelatinization. The procedure for

determining the percent solubles is given below. Ten grams of ground sample (dry matter) were added to 180 ml distilled water in a 250 ml centrifuge bottle. The contents were stirred for thirty minutes with a magnetic stirring bar which was removed and washed with an additional 10 ml distilled water. The solution was centrifuged (20° C) for fifteen minutes at 980 x G^2 (3000 rpm) using a Beckman Model J-21 Refrigerated Centrifuge. Two 10 ml aliquots of supernatant were each pipeted into a 50 ml beaker containing approximately 3 grams of Filter Flow 160 which had previously been dried for one hour at 130° C and the weight determined. Beakers containing the samples were then dried in an air oven for three hours at 130° C, after which they were cooled for two hours in a desicator and then reweighed. From the increase in weight, the increase in percent of solubles can be calculated.

Relative Spectral Reflectance

The relative spectral reflectance is an indication of the color of a product. In this study relative browning taking place during extrusion was measured. The instrument used to record the relative reflectance was an Agtron Model 500 A Relative Spectral Reflectometer. Wavelengths used were 436 nm (blue) and 540 nm (green). Products which are light red, green and yellowish are usually investigated for green reflectance. The blue mode is ideal for detecting yellow hues.

The instrument was allowed to warm up for one hour. Standard reflectance discs were selected to allow greatest use of the scale throughout all the samples to give the maximum accuracy. The reference disc used for zero and one hundred are numbers 68 and 90 at 436 nm and numbers 44 and 75 at 540 nm.

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[2]Force at center of the centrifuge bottle.

Amylograms

The amylograph cycle started at 30° C; heating continued at the rate of 1.5° C per minute for 45 minutes, with the temperature reaching 95° C. At the end of this time the gelatinized starch may be cooled at the rate of 1.5° C per minute until 50° C is reached. In this study, initial and maximum viscosity were recorded. A partially gelatinized starch sample will absorb a great deal of water and it was not possible to use the standard 100 gms of material in 450 mls water. To avoid many of the problems associated with mixing a gelatinized product, a sample of 9% solids was used. This amount of material usually placed the curve below 500 BU on the graph.

Density

Three methods were attempted to determine density: rape seed displacement, water displacement, and calculation from existing data. The rape seed displacement technique proved to be unsatisfactory due to the amount of variation produced when measuring a small volume with relatively large seeds.

The second method, water displacement, also proved to be unsatisfactory. Each sample, after being weighed, was immersed in paraffin before being placed in a water-filled graduated cylinder. One major problem with this technique is that the paraffin did not completely seal the sample, allowing water to penetrate and solubilize part of the sample.

The method selected for density determination was calculation. The mean diameter for each sample had been taken previously for expansion and by measuring the length and weight of each sample, the density could be calculated The weight of each sample was adjusted to zero percent moisture for comparison.

Microscopy

Selected samples were viewed using a light microscope and a plane polarized light microscope. A grant from the Kansas State Agriculture Experiment Station provided funds for scanning electron microscope observations and photographs.

Dextranization

Paper chromatography was used to separate short chain carbohydrates formed during extrusion. A 100 mg sample was dissolved in water and the protein percipitated with trichloroacetic acid (T.C.A.). The sample was then passed through a deionizing resin to remove interfering ions. Samples were then either spotted on Whatman number 4 paper or digested with glucoamylase. The chromatogram was developed with a solution of N-butanol, glacial acetic acid and water (4:1:1) for forty-eight hours.

Output

The output of the extruder was determined by collecting a sample for one minute. Each sample was weighed and its weight adjusted to a dry matter basis for statistical analysis.

RESULTS AND DISCUSSION

Statistical Analysis of Results

Two different computer programs were used for statistical analyses. Because of missing data points, a least squares analysis was completed. For comparison, missing data points were estimated statistically and results analyzed by an Analysis of Variance. Both analyses were computed at the Kansas State University Computing Center on an IBM Model 360 computer. Significant differences were reported at the 5% probability level. Analyses of variance were computed for each variable: moisture, temperature, shear rate, and moisture-temperature interaction.

Moisture

Moisture level of extruded wheat flour was significantly affected (5% level) only by temperature of extrusion. At the 10% level of significance, initial moisture may affect product moisture. At no reasonable level were shear rate and moisture-temperature interaction significant (Table III).

Moisture in the final product was found to be inversely related to temperature of extrusion. As final product temperature increased, there was a decrease in product moisture content (Fig. 2). Mean moisture level at each temperature was significantly different (at 5% level) from every other moisture mean (Table IV). As could be expected, higher temperatures of extrusion tend to drive off more moisture.

Product Expansion

Expansion was significantly affected by both moisture and temperature at the 5% level. Shear rate and moisture-temperature interaction had no

Table III. Summary of Statistical Analysis of Product Moisture

Treatment	DF	Alpha Estimator
Moisture	3	0.0778
Temperature	3	0.0000*
Shear Rate	2	0.2584
Moisture-Temperature	9	0.1616

*Significant at 5% level.

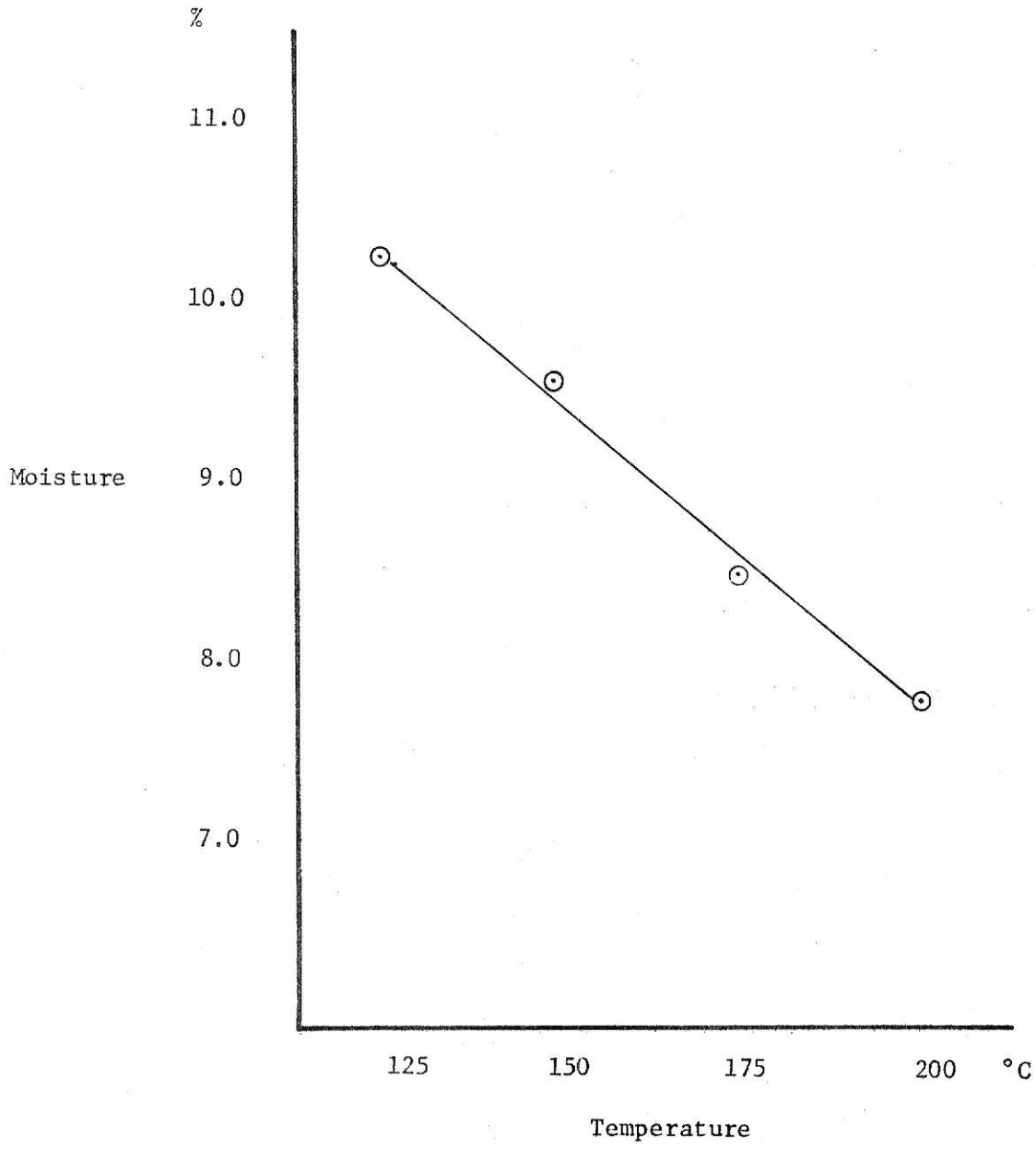

Fig. 2. Product moisture vs. temperature.

Table IV. Product Moisture Due to Temperature

LSD of Temperature Treatment 0.5344 5% Level

LSD Table

Treatment	Means	X-4	X-3	X-2	X-1
$Temperature_1$ (125°C)	10.2666	2.4833*	1.7833*	0.7000*	0.0000
$Temperature_2$ (150°C)	9.5666	1.7833*	1.0833*	0.0000	
$Temperature_3$ (175°C)	8.4833	0.7000*	0.0000		
$Temperature_4$ (200°C)	7.7833	0.0000			
Mean		T_1	T_2	T_3	T_4
		10.2666	9.5666	8.4833	7.7833
Non-significant Groupings**					

*5% level of significance.

**All are significantly different.

significance at any reasonable level (Table V).

It was found that a sample having a moisture level of 18% had the largest mean expansion. The largest single sample, however, was one with an initial moisture level of 15.5%. As can be seen in Table VI, each moisture mean was not significantly different from the next, either greater or smaller.

Expansion due to temperature was found to be significant at the 5% level (Table VII). The mean temperature with the largest expansion was 150° C. It was at this temperature that the single sample of greatest expansion was extruded. At temperatures below 150°C, there was apparently not enough energy in the system to expand the material. Above the optimum temperature, moisture was driven off and cellular structure disrupted sufficiently to reduce expansion (Fig. 3).

Shear Resistance

Resistance to shear is a relative value measured on the Brabender Extensagraph and has not been correlated to any specific unit of measure. The resulting statistical analyses indicated that initial moisture, temperature and shear rate had a significant effect on shear resistance (Table VIII).

It was found that as the moisture level decreased, there was a great increase in the shear resistance of the extrudate (Fig. 4). Flour, when cooked under dry conditions, forms a thermo-plastic material that is quite strong. Shear resistance at the 15.5% moisture level mean is not significantly different from 16.5% moisture level. Each moisture level has no significant difference from either adjacent mean (Table IX). The additional

Table V. Summary of Statistical Analysis of Expansion

Treatment	DF	Alpha Estimator
Moisture	3	0.00138*
Temperature	3	0.00072*
Shear Rate	2	0.18581
Moisture-Temperature	9	0.76397

*Significant at 5% level.

Table VI. Expansion Due to Moisture

LSD of Moisture Treatment 1.0085 5% Level

LSD Table

Treatment	Means	X-4	X-2	X-1	X-3
$Moisture_3$ (18.0%)	4.6944	2.0500*	1.6203*	0.8499	0.0000
$Moisture_1$ (15.5%)	3.8445	1.2001*	0.7704	0.0000	
$Moisture_2$ (16.5%)	3.0741	0.4297	0.0000		
$Moisture_4$ (20.0%)	2.6444	0.0000			
Mean		M_3	M_1	M_2	M_4
Non-significant Groupings					

*5% level of significance.

Table VII. Expansion Due to Temperature

LSD of Temperature Treatment 1.0085 5% Level

LSD Table

Treatment	Means	X-1	X-4	X-3	X-2
$Temperature_2$ (150°C)	4.5070	2.2049*	1.0918*	0.2740	0.0000
$Temperature_3$ (175°C)	4.0330	1.7369*	0.6178	0.0000	
$Temperature_4$ (200°C)	3.4152	1.1131*	0.0000		
$Temperature_1$ (125°C)	2.3021	0.0000			
Mean		T_2	T_3	T_4	T_1
Non-significant Groupings		———	———		

*5% level of significance.

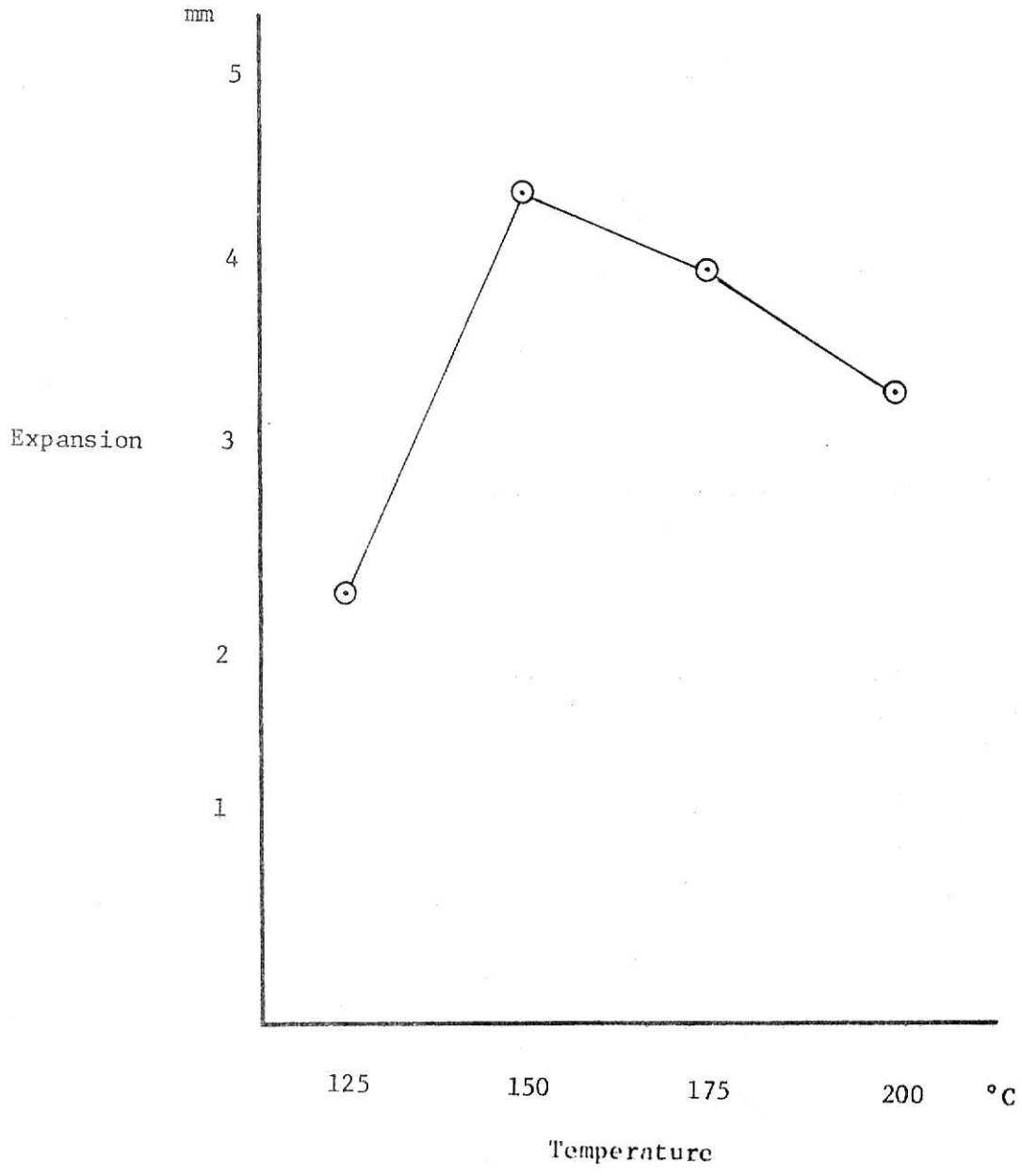

Fig. 3. Expansion vs. temperature of extrusion.

Table VIII. Summary of Statistical Analysis of Shear Resistance

Treatment	DF	Alpha Estimator
Moisture	3	0.00433*
Temperature	3	0.00000*
Shear Rate	2	0.04850*
Moisture-Temperature	9	0.47223

*Significant at 5% level.

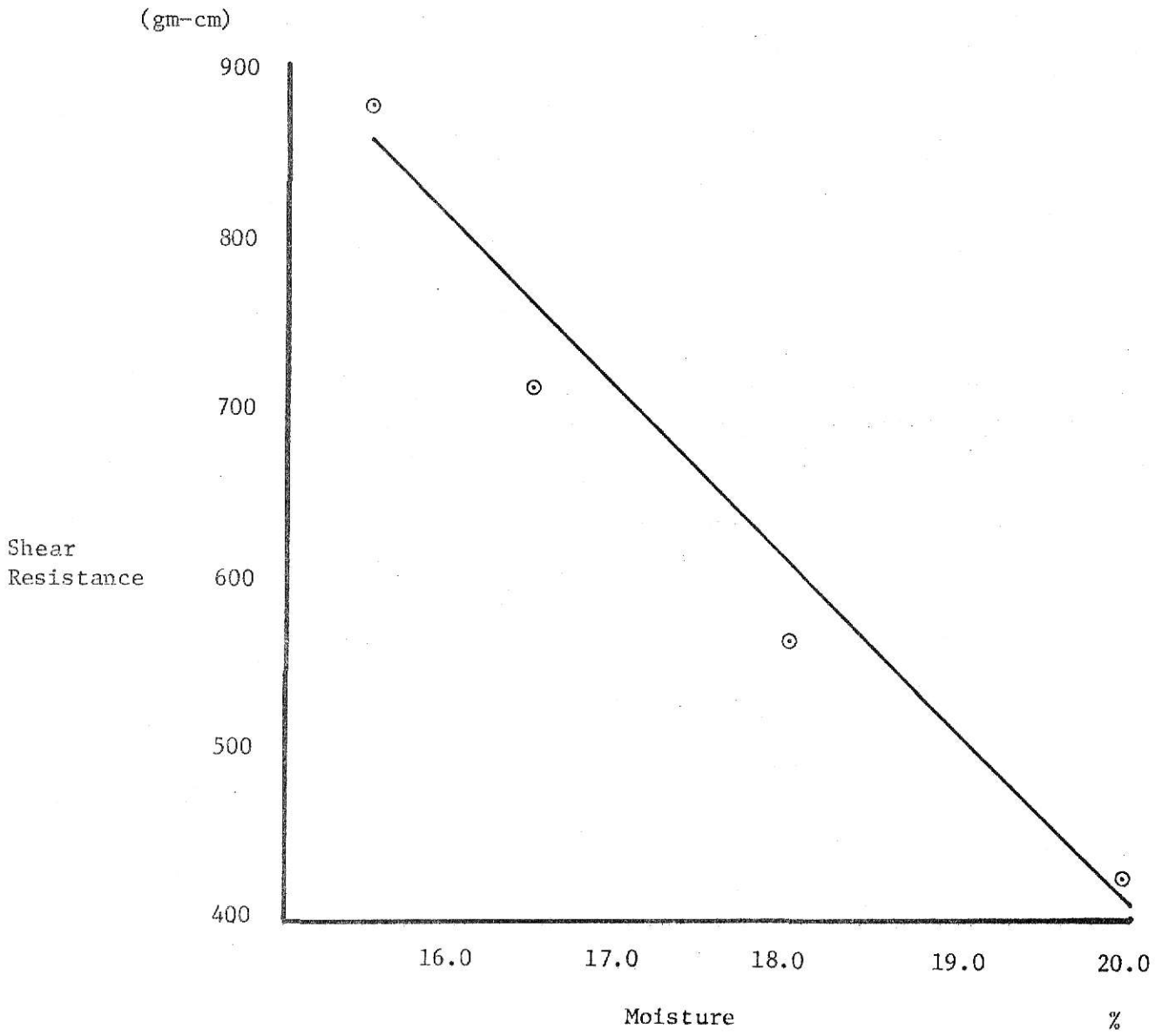

Fig. 4. Shear resistance vs. percent product moisture.

Table IX. Shear Resistance Due to Moisture

LSD of Moisture Treatment 215.1388 5% Level

LSD Table

Treatment	Means	X-4	X-3	X-2	X-1
$Moisture_1$ (15.5%)	874.1665	393.7500*	307.0833*	150.7500	0.0000
$Moisture_2$ (16.5%)	715.4165	235.0000*	148.3332	0.0000	
$Moisture_3$ (18.0%)	567.0832	86.6667	0.0000		
$Moisture_4$ (20.0%)	480.4165	0.0000			
Mean		M_1	M_2	M_3	M_4
Non-significant Groupings					

*5% level of significance.

moisture in the system provided a greater number of sites where gas cells may be formed. As the moisture is heated to steam, the expanding gas tends to disrupt the carbohydrate structure, thereby reducing its resistance to shear.

Temperature of extrusion was also found to have a significant difference in mean (Table X). There are two temperatures that did not show significant differences, 125° C and 150° C. This is most probably due to the fact that it was not possible to maintain 125° C throughout the sampling due to the frictional heat developed. It can be seen from Fig. 5 that as temperature increased there was a reduction in shear resistance. This was the result of the thermal degradation of starch and protein structures at high temperatures.

The third factor to show significant differences between the means of shear resistance is the shear rate at which samples were produced. Shear rate can be correlated to two other factors: mixing action and retention or cooking time. As shear rate increases, mixing action inside the barrel of the extruder becomes more vigorous and greater physical damage can occur. At the higher shear rates, time of product exposure to cooking temperatures is reduced. The faster shear rates and their destructive action tend to reduce the strength of the product (Fig. 6). As seen in Table XI, the slower shear rates provided for a stronger product. Destructive action of the screw inside the barrel appears to have a more significant effect than does possible greater thermal degradation at lower shear rates.

Table X. Shear Resistance Due to Temperature

LSD Temperature Treatment 215.1388 5% Level

LSD Table

Treatment	Means	X-4	X-3	X-2	X-1
$Temperature_1$ (125°C)	1062.0830	925.9164*	521.2498*	137.1833	0.0000
$Temperature_2$ (150°C)	924.9997	815.8221*	384.1665*	0.0000	
$Temperature_3$ (175°C)	540.8332	431.6666*	0.0000		
$Temperature_4$ (200°C)	109.1666	0.0000			
Mean		T_1	T_2	T_3	T_4
Non-significant Groupings		———			

*5% level of significance.

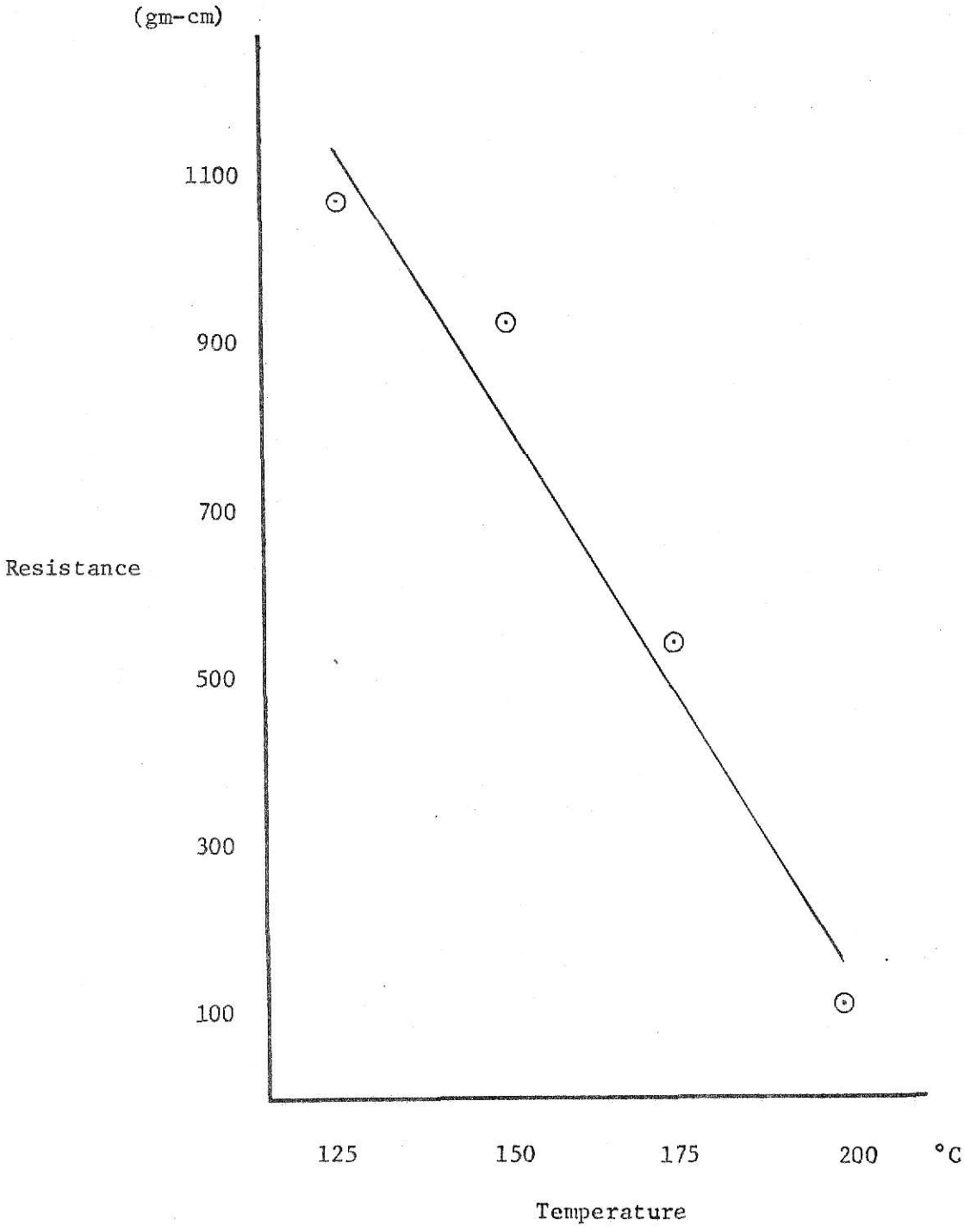

Fig. 5. Shear resistance vs. temperature.

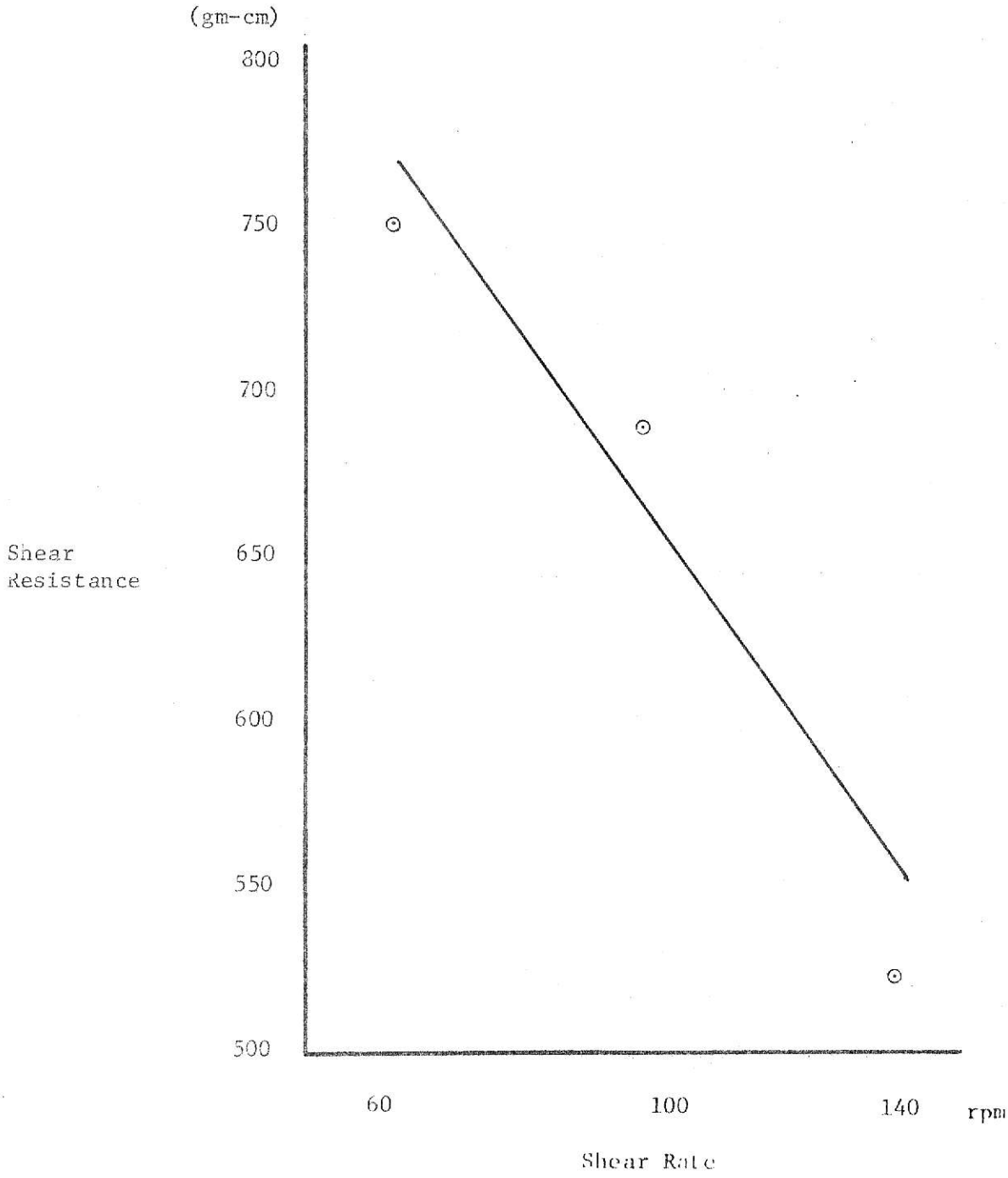

Fig. 6. Shear resistance vs. shear rate.

Table XI. Shear Resistance Due to R.P.M.

LSD R.P.M. Treatment 186.3157 5% Level

LSD Table

Treatment	Means	X-3	X-2	X-1
RPM_1	757.5000	230.6250*	64.0625	0.0000
RPM_2	693.4375	166.5625	0.0000	
RPM_3	526.8750	0.0000		
Mean		RPM_1	RPM_2	RPM_3
Non-significant Groupings				

*5% level of significance.

Increase in Solubles

The increase in solubles can be related to degree of gelatinization caused by thermal and physical damage during extrusion. The only parameter that caused a significantly different increase in solubles was temperature (Table XII). As the temperature increased, so did the amount of solubles in a nearly linear relationship (Fig. 7). The other parameters, moisture, shear rate, and moisture-temperature interaction, did not show any significantly different means at the 5% level (Table XIII). It should be noted that moisture becomes significant if the protection level for a type I error is reduced to the 7.9% level. At no reasonable production level did shear rate and moisture-temperature interaction show any significantly different means.

Relative Spectral Reflectance

The color of each sample was determined by the amount of reflectance obtained. Lower reflectance values indicate that samples reflect less electromagnetic radiation and had more color than a sample with a high reflectance value. The only source of color for these samples was the browning that took place during the extrusion process. It is not surprising, therefore, that temperature and temperature-related effects exhibited significant differences. As seen in Table XIV and Table XV, the alpha estimators indicate that temperature, shear rate, and moisture-temperature interaction would be significant at less than the 1% protection level.

Color due to temperature treatment for both wavelengths used increased as temperature increased. Separation of means indicates that at 200° C mean reflectance was significantly different from all others at both

Table XII. Increased Solubles Due to Temperature

LSD of Temperature Treatment 3.1609 5% Level

LSD Table

Treatment	Means	X-1	X-2	X-3	X-4
$Temperature_4$ (200°C)	25.1420	14.8872*	7.7369*	3.8639*	0.0000
$Temperature_3$ (175°C)	21.2781	11.0183	3.8730*	0.0000	
$Temperature_2$ (150°C)	17.4051	7.1453*	0.0000		
$Temperature_1$ (125°C)	10.2598	0.0000			
Mean		T_4	T_3	T_2	T_1

Non-significant Groupings**

*5% level of significance.

**All means are significantly different.

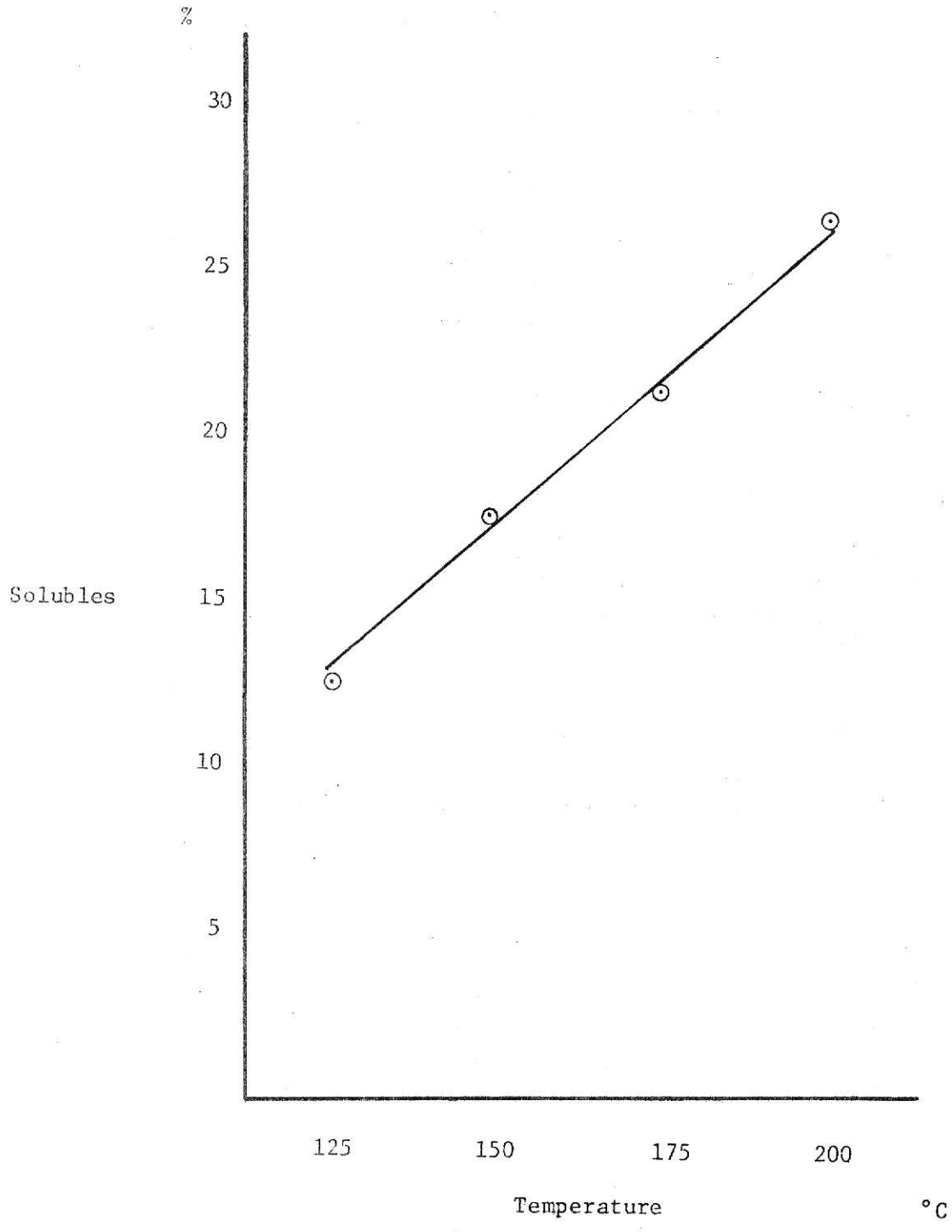

Fig. 7. Percent increased solubles vs. temperature.

Table XIII. Summary of Statistical Analysis of Increased Solubles

Treatment	DF	Alpha Estimator
Moisture	3	0.07815
Temperature	3	0.00000*
Shear Rate	2	0.85376
Moisture-Temperature	9	0.18103

*Significant at 5% level.

Table XIV. Summary of Statistical Analysis of Relative Spectral Reflectance (436 nm)

Treatment	DF	Alpha Estimator
Moisture	3	0.05345
Temperature	3	0.00000*
Shear Rate	2	0.00954*
Moisture-Temperature	9	0.00259*

*Significant at 5% level.

Table XV. Summary of Statistical Analysis of Relative Spectral Reflectance (540 nm)

Treatment	DF	Alpha Estimator
Moisture	3	0.06169
Temperature	3	0.00000*
Shear Rate	2	0.02534*
Moisture-Temperature	9	0.00212*

*Significant at 5% level.

wavelengths used (Table XVI and Table XVII). Figure 8 gives an indication of the difference of reflectance between the two wavelengths--each pair of points is separated by six units.

For both wavelengths used, shear rate was a contributing factor to product color. At lower shear rates, the material had more time to be affected by the heat of the extruder barrel. The fastest shear rate did not show significant difference from the intermediate but was significantly different from the lowest shear rate (Table XVIII and Table XIX).

Significant interaction of moisture and temperature was probably most affected by temperature. As the temperature increased, less moisture that could absorb heat remained in the system. At lower moisture levels and high temperatures, the extrudate usually exhibited the lowest reflectance.

Amylograph Viscosity

The only treatment that did not give significantly different means was shear rate. The other treatments, moisture, temperature, and moisture-temperature interaction, gave significantly different means at the 5% level (Table XX).

This study indicates that the more moisture available, the greater the disruption of starch granules when interacted with heat. Moisture at room temperature will generally not significantly affect the starch granule. Interaction of moisture and temperature was the significant factor affecting amylograph viscosity after five minutes stirring time.

Table XVI. Relative Spectral Reflectance

Due to Temperature (436 nm)

LSD of Temperature Treatment 8.6653 5% Level

LSD Table

Treatment	Means	X-4	X-3	X-2	X-1
$Temperature_1$ (125°C)	81.4666	35.8832*	14.1333*	6.3416	0.0000
$Temperature_2$ (150°C)	75.1249	29.5416*	7.7916	0.0000	
$Temperature_3$ (175°C)	67.3333	21.7499	0.0000		
$Temperature_4$ (200°C)	45.5833	0.0000			
Mean		T_1	T_2	T_3	T_4
Non-significant Groupings					

*Significant at 5% level.

Table XVII. Relative Spectral Reflectance

Due to Temperature (540 nm)

LSD of Temperature Treatment 8.3799 5% Level

LSD Table

Treatment	Means	X-4	X-3	X-2	X-1
$Temperature_1$ (125°C)	72.4999	35.0416*	10.1666*	2.7500	0.0000
$Temperature_2$ (150°C)	69.7499	32.2916*	7.4166	0.0000	
$Temperature_3$ (175°C)	62.3333	24.8750*	0.0000		
$Temperature_4$ (200°C)	37.4583	0.0000			
Mean		T_1	T_2	T_3	T_4
Non-significant Groupings					

*Significant at 5% level.

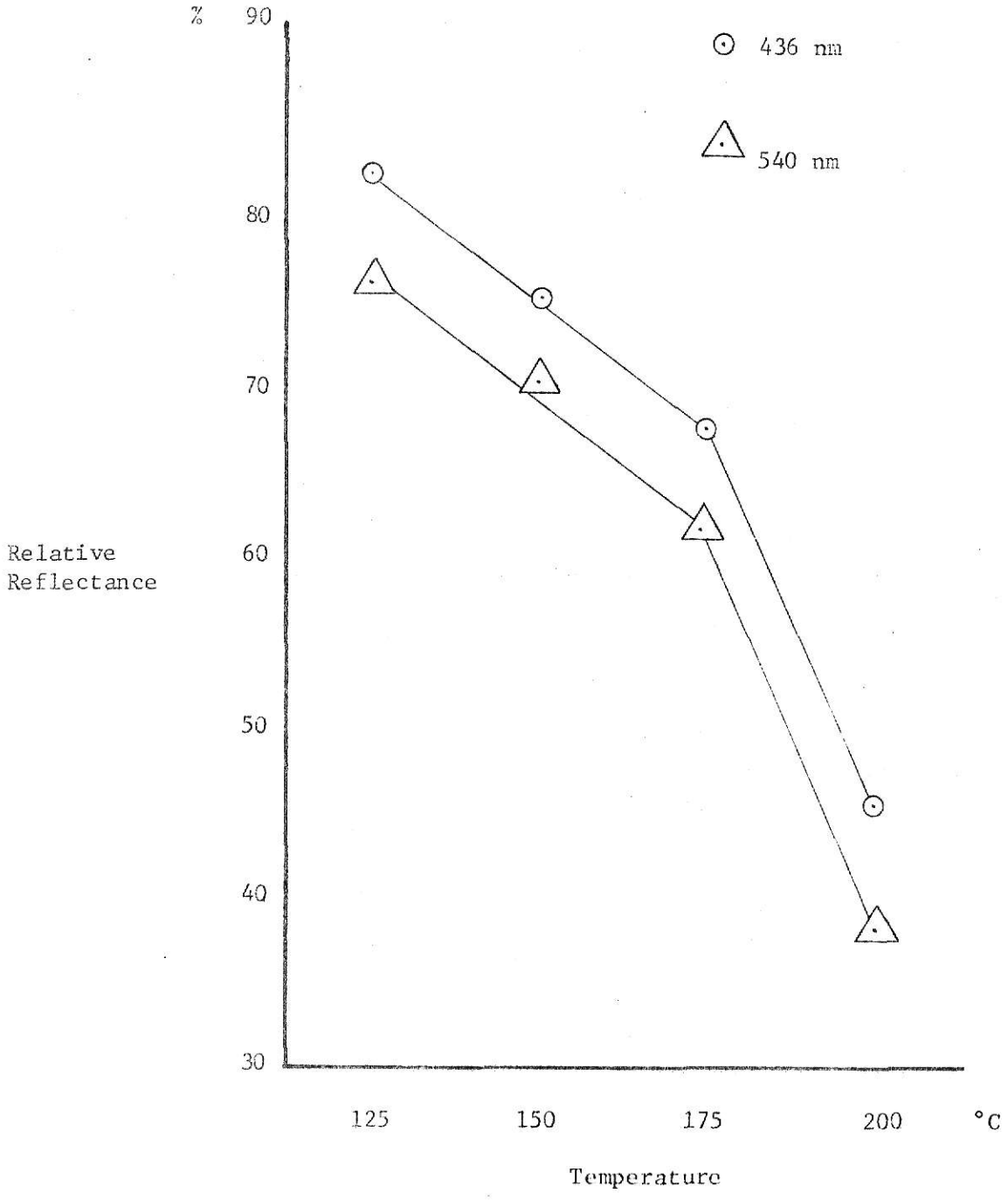

Fig. 8. Relative spectral reflectance vs. temperature.

Table XVIII. Relative Spectral Reflectance

Due to RPM (436 nm)

LSD of RPM Treatment 7.5048 5% Level

LSD Table

Treatment	Means	X-1	X-2	X-3
RPM_3 (140)	73.8125	12.0562*	7.2500	0.0000
RPM_2 (100)	66.5625	4.8062	0.0000	
RPM_1 (60)	61.7562	0.0000		
Mean		73.8125	66.5025	61.7562

Non-significant Groupings

*5% level of significance.

Table XIX. Relative Spectral Reflectance

Due to RPM (540 nm)

LSD of Shear Rate Treatment 7.2572 5% Level

LSD Table

Treatment	Means	X-1	X-2	X-3
Shear Rate$_3$ (140 rpm)	65.8750*	10.2187	5.8750	0.0000
Shear Rate$_2$ (100 rpm)	60.0000*	4.3437	0.0000	
Shear Rate$_1$ (60 rpm)	55.6562*	0.0000		
Mean		S_3	S_2	S_1
Non-significant Groupings				

*Significant at 5% level.

Table XX. Summary of Statistical Analysis of Amylograph Viscosity (5 min)

Treatment	DF	Alpha Estimator
Moisture	3	0.01978*
Temperature	2	0.00000*
Shear Rate	2	0.63781
Moisture-Temperature	9	0.01549*

*Significant at 5% level.

Amylograph Peak Viscosity

Treatments that most affected peak amylograph viscosity were temperature and moisture-temperature interaction. At no reasonable alpha level did shear rate and moisture become significant (Table XXI). There were apparently no significant differences between the lower temperatures. At a temperature of 200°C, at all moisture levels and shear rates, there was sufficient thermal degradation to significantly reduce peak viscosity of the amylograph (Table XXII). Moisture-temperature interaction significance indicated that the greater the amount of moisture in the system being heated to hotter temperatures, the greater was the degree of gelatinization.

Density

Density was calculated and recorded in the units grams per cubic centimeter (gm/cc). The only treatment to cause significantly different means was temperature. Other treatments would not give significantly different means at any reasonable alpha level (Table XXIII).

As could be expected, increased temperature caused a decrease in density of the product (Table XXIV). As temperatures reached the upper range (175 to 200°C), the difference became less to a point of being non-significant. Higher temperatures produced a product that was much lighter than at lower temperatures.

Extruder Output

The amount of material the extruder expelled per minute was determined and the only treatment that gave significantly different means was the shear rate (Table XXV). Moisture, temperature, and moisture-temperature

Table XXI. Summary of Statistical Analysis of Peak Amylograph Viscosity

Treatment	DF	Alpha Estimator
Moisture	3	0.37264
Temperature	3	0.00210*
Shear Rate	2	0.46421
Moisture-Temperature	9	0.00002*

*Significant at 5% level.

Table XXII. Amylograph Peak Viscosity Due To Temperature

LSD Table

Treatment	Means	X-4	X-3	X-2	X-1
$Temperature_1$ (125°C)	367.4997	85.4165*	14.1665	2.5000	0.0000
$Temperature_2$ (150°C)	364.9997	82.9165*	11.6665	0.0000	
$Temperature_3$ (175°C)	353.3332	71.2000*	0.0000		
$Temperature_4$ (200°C)	282.0832	0.0000			
Mean		T_1	T_2	T_3	T_4
Non-significant Groupings		———	———		

*Significant at 5% level.

Table XXIII. Summary of Statistical Analysis of Density

Treatment	DF	Alpha Estimator
Moisture	3	0.30184
Temperature	3	0.00000*
Shear Rate	2	0.53429
Moisture-Temperature	9	0.92906

*Significant at 5% level.

Table XXIV. Density Due to Temperature

LSD of Temperature Treatment 0.1598 5% Level

LSD Table

Treatment	Means	X-4	X-3	X-2	X-1
$Temperature_1$ (125°C)	0.8374	0.7161*	0.6745*	0.4721*	0.0000
$Temperature_2$ (150°C)	0.3653	0.2440*	0.2024*	0.0000	
$Temperature_3$ (175°C)	0.1629	0.0415	0.0000		
$Temperature_4$ (200°C)	0.1213	0.0000			
Mean		T_1	T_2	T_3	T_4
Non-significant Groupings				———	———

*Significant at 5% level.

Table XXV. Summary of Statistical Analysis of Extruder Output

Treatment	DF	Alpha Estimator
Moisture	3	0.07519
Temperature	3	0.22213
Shear Rate	2	0.00000*
Moisture-Temperature	1	0.11826

*Significant at 5% level.

interaction had no significant differences. The faster the extruder screw turns, the more material that passes through the machine.

Microscopy

When viewed under a light microscope, products appeared to contain intact starch granules. Products that were extruded at lower temperatures and high moisture levels had more granules or parts of granules that would absorb iodine than samples cooked at higher temperatures. When viewed under plane polarized light, no starch granules exhibited birefringence. This fact indicates that starch structure had been altered.

The scanning electron micrographs (Plate I) are of a lower moisture sample (top) and a high moisture sample (bottom). A marked difference in cell wall structure of the two samples can be seen. In the lower moisture sample, the cell wall structure has a much thicker wall and the cell size is much smaller. This type of structure gives a very strong, rigid product. The higher moisture samples were extruded at a higher temperature which helped give rise to the lighter, fluffier product. The additional sites for cells to form, along with the necessary heat for expansion, are the reasons for this type of product.

Plate II shows a sample (top) cross-section at a relatively low magnification (44X). The lower micrograph is a high magnification (1900X) of the internal surface of one of the cells above. Seen on the surface are two lobes that have a checked appearance. These fissures are artifacts caused by the heating with a focused electron beam. The result was a swelling and fracture in the gold coating that could have been caused by a portion of a starch granule swelling. This action was much the same as when heating starch on a hot stage microscope.

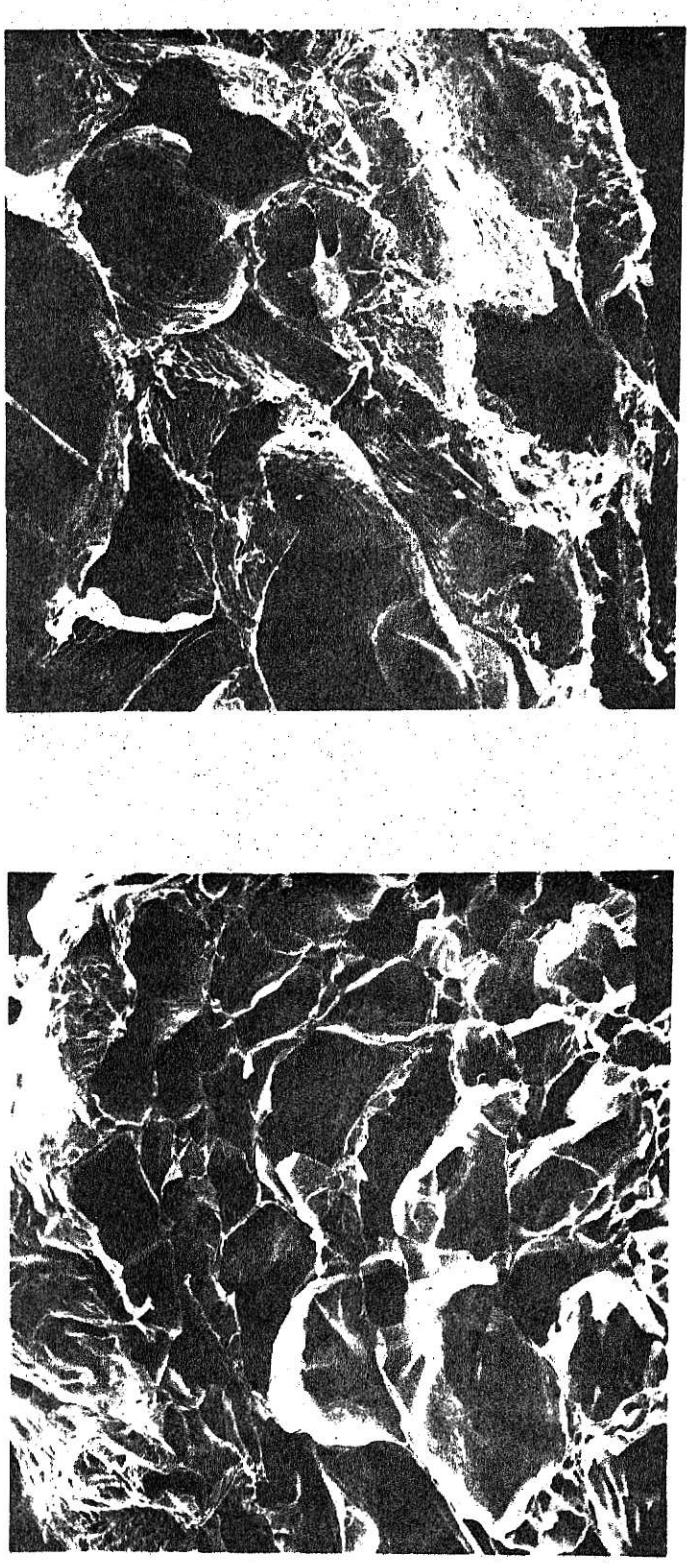

Plate I. A low moisture extrudate (top). A high moisture extrudate (bottom).

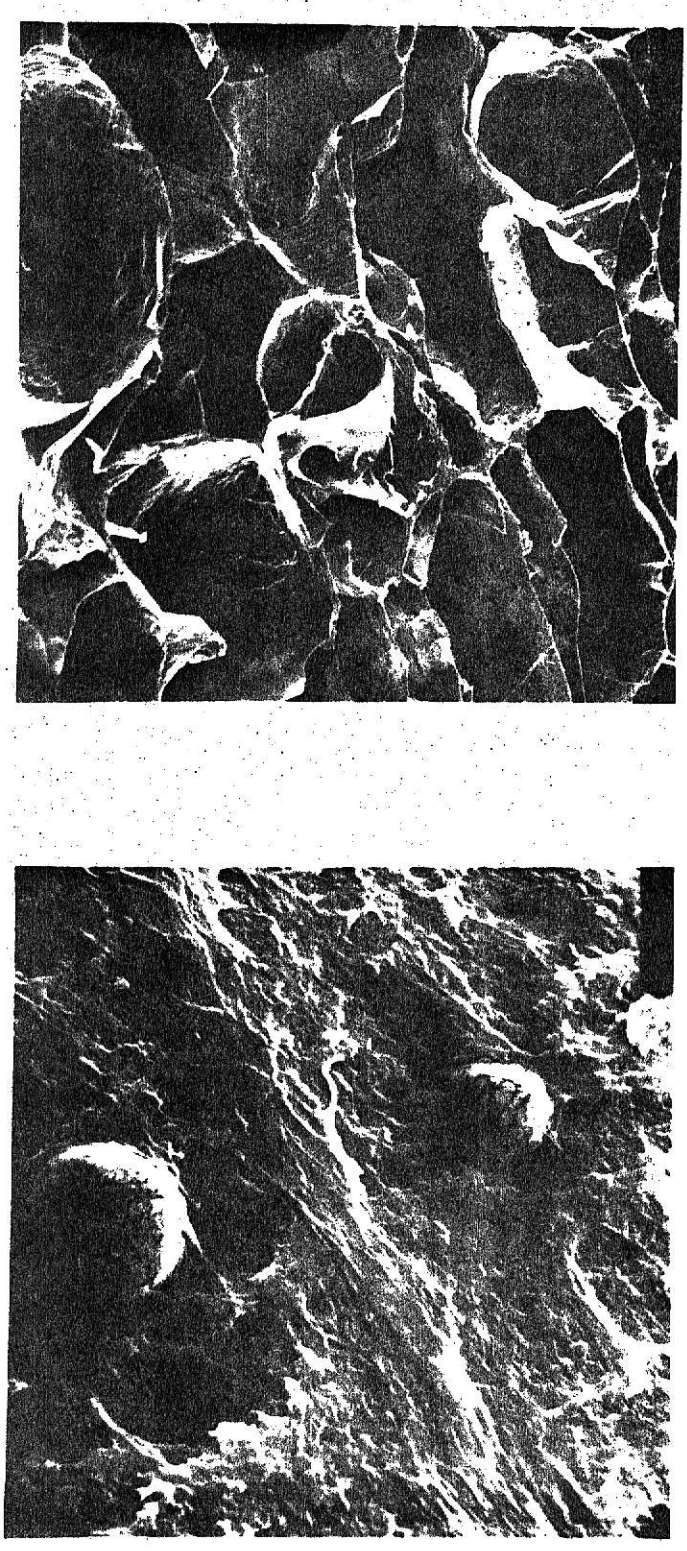

Plate II. Low magnification of extrudate (top). High magnification of extrudate (bottom).

Dextranization

Chromatograms of two samples are shown in Plate III, a high moisture sample, and Plate IV, a low moisture sample. Column one on each plate is the sample without enzyme digestion. The other two columns have been digested with glucamylase for thirty minutes (column two) and sixty minutes (column three). From these chromatograms, it can be seen that there are more short chain dextrine formed under the drier conditions, temperatures being equal.

The large percentage of glucose upon digestion with the enzyme indicates that no unusual bonds were formed during extrusion.

Two characteristics that were not analyzed because of lack of data are the power requirements and the protein level of the flour. It was generally noticed that the power required to extrude wheat flour increased with the lowering of the moisture level. As the temperature was increased, some drop in power requirements was noted. The protein level of the flour seemed to affect the ability of the machine to extrude the flour. The sample of flour used had a protein level of 10.9%. One sample of flour containing 14.2% protein was tried but was incapable of being extruded with the equipment used.

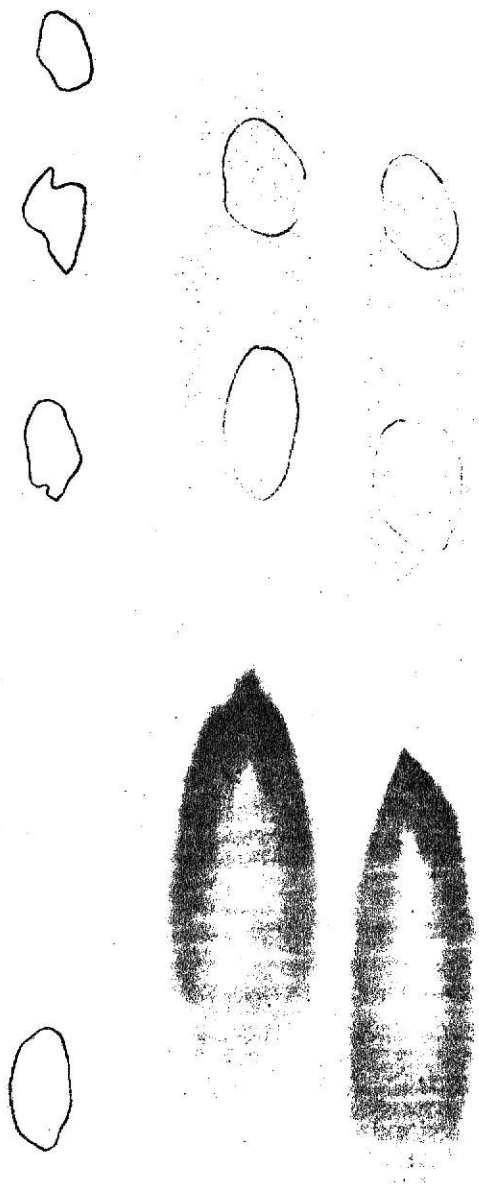

Plate III. Enzyme degradation of high moisture sample.

Plate IV. Enzyme degradation of low moisture sample.

SUMMARY AND CONCLUSIONS

Purposes of this investigation were to determine some of the effects of extrusion cooking on wheat flour, to measure some of the physical characteristics of the extrudate and to learn something about the gelatinization of the starch. The three variables chosen for study were four initial moisture levels, four temperatures, and three shear rates.

It was found that product moisture level is not significantly affected by the initial moisture level of the uncooked flour at the moisture levels examined. The one factor that does appear to affect moisture level is the temperature at which extrusion was performed.

Expansion of the product is influenced significantly by moisture level of the flour and temperature of extrusion. Shear rate and moisture-temperature interaction do not appear to make significant contributions to expansion. It appeared that temperature must reach a minimum level before significant expansion occurs. As the temperature is increased, however, breakage of cellular structure occurs and expansion decreases.

Shear resistance was not significantly affected by moisture-temperature interaction. All other variables have significant differences. Low moisture samples extruded form a thermo-plastic material that is resistant to breaking forces. As moisture increases, there is a marked decrease in force required to break a sample. Temperature during extrusion is also important to strength. As temperature increases, there is more thermal degradation of product, hence reduced shear resistance. Shear rate is related to time of cooking and the more a product has been cooked, the less resistant to shear it becomes.

Solubles present in a flour solution can give some indication as to the disruption and degradation the starch has undergone. Here again, temperature is the significant factor. The higher the temperature, the more soluble the product.

Color of the product is related to temperature. As the temperature is increased, so is the color. Shear rate is also a significant parameter in that the longer the cook, the more color that is formed. The two wavelengths used (436 nm and 540 nm) gave results that were very similar.

Amylograph viscosity after stirring five minutes was affected least by shear rate. Moisture, temperature, and moisture-temperature interaction are the parameters of significance. It is clearly seen that as moisture and temperature increase, the amount of gelatinized starch increases.

Density of the product was significantly affected by temperature. Lower temperatures produced a strong, dense product, whereas high temperature resulted in a light, fluffy product.

The outflow of product (in grams per minute) is related to shear rate. The faster the extruder turns, the more product that is produced.

From electron micrographs it was learned that cellular structure becomes larger and cell walls thinner with increased temperature. There were no artifacts or objects that resembled an intact starch granule seen.

In every examination except outflow, temperature was the treatment, or one of the treatments, that significantly affected the results. It may be concluded, therefore, that temperature is possibly the most important factor of extrusion. Moisture contained in the raw material or that is added before extrusion was the second most important factor. A valid

assumption about temperature or moisture cannot be made without consideration of the other parameter. When moisture levels are increased, more heat is lost from the system, producing steam. At the lower limits of moisture (15.5% under these conditions), the product has been cooked to a much greater degree.

It is possible to predict, to some extent, the type of product that might be expected when extruding wheat flour with this type of equipment under these parameters. For a low density product that has little resistance to shear, and a high solubles content, high temperature and moisture are needed. For a light, but strong product, a decrease in moisture is needed. For a hard, dense product, a low temperature system is used.

ACKNOWLEDGEMENTS

The author is greatly indebted to Professor John A. Johnson for his help and supervision of the study and guidance in the preparation of this manuscript.

Gratitude is expressed to Dr. W. J. Hoover, Head of the Department of Grain Science and Industry, for the provision of research facilities and to other members of the staff of the Department of Grain Science and Industry and to Mark Stearns, graduate student, Department of Grain Science and Industry, without whose help the collection of samples would have been very difficult.

Acknowledgement is given to the Quaker Oats Company for their financial assistance to complete this study.

LITERATURE CITED

1. Anderson, R. A., Conway, H. F., Pfiefer, V. R. and Griffin, E. L.
Gelatinization of Corn Grits by Roll and Extrusion Cooking
Cereal Sci. Today 14(1):4(1969).

2. Anderson, R. A., Pfiefer, V. F., Bookwalters, G. N. and Griffin, E. L.
Instant Corn-Milk-Soy Food Blends for World Wide Feeding
Cereal Sci. Today 16(1):5(1971).

3. Anker, C. A. and Geddes, W. F.
Gelatinization Studies Upon Wheat and Other Starches
Cereal Chem. 21:335(1944).

4. Association of Official Agricultural Chemists.
Official Methods of Analysis (11 ed.), p. 212.
The Association: Washington, D. C. (1970).

5. Conway, H. F.
Extrusion Cooking of Cereals and Soy Beans: Part II
Food Prod. Develop. 5(2):27(1971).

6. Griff, A. L.
Plastics Extrusion Technology, 2 ed.
Van Nostrand-Reinhold Co., (1968).

7. Harper, J. M.
Research Needs in Extrusion Cooking and Forming
Presentation to Am. Soc. Agri. Engs. Annual Meeting
December 7, 1971.

8. de Muellenaere, H. J. H. and Buzzard, J. L.
Cooker Extrusion in Service to World Feeding
Food Tech. 23:345(1969).

9. Murphy, E. J.
Thermal Degradation of Carbohydrates, A Review
J. Agri. Food Chem. 17(4):747(1969).

10. Mustachas, G., Griffin, F.,Allen, L. and Smith, O.
Production and Nutritional Evaluation of Extrusion--Cooked Full-Fat Soybean Flour
J. Am. Oil. Chem. Soc. 41(9):607(1964).

11. Pomeranz, Y., ed.
Wheat Chemistry and Technology, 2 ed.
American Association of Cereal Chemists 1971
The Association, St. Paul, Minnesota.

12. Pyler, E. J.
Baking Science and Technology, Vol. I, p. 17
Seiber Publishing Co. (1952).

13. Reeve, R. M. and Walker, H. G.
The Microscopic Structure of Popped Cereals
Cereal Chem. 46:227(1969).

14. Sanderude, K. G.
Continuous Cooking Extrusion: Benefits to the Snack Food Industry
Cereal Sci. Today 14(6):210(1969).

15. Sanderude, K. G.
Extrusion Cooking--Principles, Applications for the Snack Food Industry
Wenger Mixer Mfg. Co.

16. Sanderude, K. G.
Continuous Cooking of Cereal Grains and Wheat Flours by Short Time-High Temperature Extrusion
Northwestern Miller, 276(6):21(1969).

17. Sanderude, K. G.
Food Engineering--Continuous Cooking Extrusion of Cereal Grains in the Food and Feed Industries
Presentation to the 60th Annual Meeting American Society of Agricultural Engineers in Joint Meeting with Canadian Society of Agricultural Engineers. June 27, 1967.

18. Sanderude, K. G.
Needs, Uses, and Basic Procedures for Continuous Cooking Extrusion in the Food Industry
Presentation to the 55th Annual Meeting of the American Association of Cereal Chemists, October 20, 1970.

19. Sanderude, K. G., Ziemba, J. V.
New Products Come Easy with Extrusion Cooking
Food Eng. 40(8):67(1968).

20. Sandsted, R. M. and Abbot, R. C.
A Comparison of Methods for Studying the Course of Starch Gelatinization
Cereal Sci. Today 9(1):13(1964).

21. Smith, O. B.
Pre-Cooking of Cereal and Protein Blends
Presentation to the International Symposium on Oilseed Proteins and Concentrates, July 3, 1967.

22. Watson, C. A. and Johnson, J. A.
Studies on Gelatinization of Starch. Competition for Water by Proteins.
J. Food Sci. 3(3):450(1965).

23. Williams, M. A. and Baer, S.
The Effects of Expansion Upon Cereal Grains and Oilseeds
Bulletin No. 111.025
The V. O. Anderson Co., Cleveland, Ohio.

24. Williams, M. A. and Baer, S.
Introduction to Grain Expansion
Presentation to the American Society of Agricultural Engineers,
December 7, 1965.

CHARACTERIZATION OF EXTRUDED WHEAT FLOUR

by

WAYNE R. MOORE

B. S., Kansas State University, 1971

AN ABSTRACT OF A MASTER'S THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Grain Science and Industry

KANSAS STATE UNIVERSITY
Manhattan, Kansas

1973

A Hard Red Winter Wheat flour was extruded using a laboratory extruder and the characteristics of the extrudate examined. Samples of 15.5%, 16.5%, 18.0%, and 20.0% moisture were extruded at 125°C, 150°C, 175°C, and 200°C using three shear rates (60rpm, 100rpm, 140rpm) for each combination of moisture and temperature. The following characteristics for each sample obtained was examined: moisture, extrudate expansion, shear resistance, increase in water solubles, color, density, amylograph viscosity, and extruder outflow. In addition, scanning electron micrographs were taken of some samples. Samples were ground on a laboratory hammer mill to pass through a 0.020 inch screen.

It was found that the three variables, at the levels observed, were interrelated. The temperature level, moisture level, and the shear rate effect the results of the others in some instances. Temperature, at the levels observed, had a significant affect on all characteristics examined. The expansion and density of the extrudate is affected, to some degree, by the moisture present that has been expanded by heating the flour. The increase in solubles is directly affected by the temperature as is the color of the resultant product.

Moisture levels of the raw material, while statistically significant in some analyses, seemed to function mainly as related to temperature. Moisture levels of the flour played a significant role in the ease of which the extruder operated; the lower moisture samples being more difficult to extrude.

Shear rate was a significant affector of the outflow. The higher the revolutions per minute, the more material that was extruded. Shear rate is related to retention time, the length of time the material is exposed to the conditions of extrusion.

Of the three parameters observed, temperature had the greatest affect on the characteristics observed.