EXTRUSION COOKING OF WHEAT STARCH

by

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INTRODUCTION

Three general types of food extrusion are used at present (1). They are low-pressure cold forming, low-pressure cooking and forming, and high-pressure cooking and forming. Cold forming is the low pressure extrusion of premixed soft masses into shaped pieces. The shaped pieces are normally further processed by deep-fat frying, drying, or baking. Low-pressure cooking and forming involves feeding dry ingredients which are moistened, mixed, and cooked as desired within the extruder barrel. The final product temperature is characteristically below the boiling point of water, thus allowing a shaping and cutting process at the extruder die or in an auxiliary piece of equipment. Those products may be further processed by frying, puffing, or toasting. High-pressure cooking and forming is characterized by sufficient heat and moisture to thoroughly cook and homogenize all the ingredients. In high-pressure extrusion cooking the material is heated to temperatures above 100°C. The elevated temperatures combined with the physical design of the extruder create high pressure and shear within the extruder barrel. When the extrudate emerges from the die, moisture flashes off as steam and the sudden drop in pressure can cause the product to expand and become porous.

One application of cold forming is dough extrusion to make products such as doughnut holes and bread sticks. Low-pressure cooking and forming is used widely for breakfast cereal production, whereas high-pressure cooking and forming is used for the manufacture of snack foods, gelatinized starches, and texturized protein products.

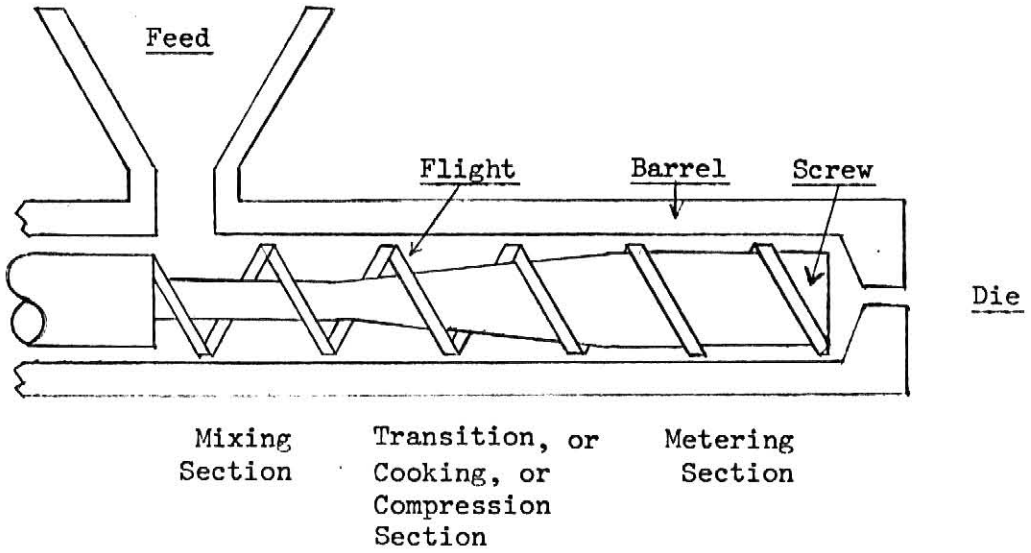
As extrusion has progressed, possibly the most important development has been that of high temperature-short time (HT-ST) extrusion cooking. HT-ST extrusion cooking is essentially high-pressure cooking and forming where the extruder is operated at temperatures and pressures which facilitate a relatively short cooking time. HT-ST extrusion cooking in the food and feed industry was originally developed for economical cooking of cereal grains. Today extruders are cooking a variety of cereal based mixtures including snacks, meat extenders, breadfast cereals, and pet and livestock feeds.

The many advantages of extrusion cooking have led to its rapid acceptance in the food industry. One such advantage is the increased digestibility obtained from complete starch gelatinization. This is important for foods or beverages which are to be marketed in a precooked form. The thermal inactivation of growth inhibitors and antipalatability factors found in certain oilseeds and pulses has led to the acceptance of extrusion cooking for the production of protein-fortified foods. The ability of extruders to change certain vegetable proteins into a texturized meat-like product has recently flooded the meat market with meat extenders and lowered the price of certain meat products. The production of a product having a low bacterial count and free from pathogens, molds, and live insects assures the extrusion processor of a sanitary product. The utilization of extruders has produced a large number of snacks and breakfast cereals in a wide variety of shapes, sizes, and textures. Those reasons coupled with the capacity of an extrusion cooker to continuously yield uniform products have contributed to its versatility and acceptance in the food industry (2).

The most popular extruder design consists simply of a close tolerance screw rotating inside a barrel. The barrel is constricted at the discharge end, A) to create a back pressure to facilitate mixing and, B) to shape the product. (See figure 1, page 4). Each piece of the extruder can be modified in different ways to cater to specific needs. The feeder may be a gravimetric feeder, a vibratory feeder, a dry force-feeder, or a feeder-conditioner combination. Screws with a varied pitch and depth of flighting can give varied shear and compression to the product. The compression of the material within an extruder may also be altered by increasing the shaft diameter of a constant pitched screw or by tapering the screw into a conical barrel. The inside of the extruder barrel may be either smooth or grooved. The grooves help in the development of frictional heat and may be of any shape or depth and straight or rifled. Straight grooves cause the development of more frictional heat than rifled grooves. The heat control system may be either steam or electrically heated and have a liquid or air-cooling system. The die which controls the shape of the product may be of any shape or size as long as continuous flow is maintained throughout the inside of the die. The size and shape of the die also has direct effect on the pressure within the extruder barrel (3).

The extrusion process begins when the raw material is fed through the feeder into the barrel. The material enters the feed zone of the barrel where moisture is often added. The material is mixed as it is conveyed down the barrel to the transition or cooking zone. In this zone the temperature of the material rises rapidly, and the product is compressed. At the end of this zone the produce is fully compressed and

Figure 1
Schematic of Extrusion Cooker



in theory, all the material has been cooked (starch fully gelatinized). The final zone of metering zone mechanically works the material into a uniform, fluid plastic-state and under high pressure conveys the material to the die. The material is then forced through the die where the sudden reduction in pressure causes the product to expand and much of the moisture to flash off as steam.

A typical formula for the production of a textured puffed snack would be 20% dry-blend texturized soy protein (55% protein), 5% wheat flour, 60% tapioca flour, and 15% seasoning such as onions or garlics (4). Approximately sixty-five percent of this formula consists of starch. This formula illustrates that starch is frequently the major constituent in extruded food products. Much of the know how and knowledge concerning the art or science of food extrusion cooking has been attained in research departments of individual companies and has not been published or made available (3). Very little work has been reported concerning the effects of high-temperature, short-time extrusion-cooking on starch. This investigation was undertaken to determine some of the physical and chemical effects of HT-ST extrusion cooking on wheat starch. Three extrusion parameters (moisture, temperature, and screw speed) were examined for their effects upon selected properties (pH, viscosity, expansion, color, water solubility, enzyme digestibility) of the starch extrudate.

Starch occurs naturally in cereal grains in a granular form. A starch granule consists almost exclusively of two, high molecular-weight D-glucose polymers. The amylose polymer is a linear molecule consisting of D-glucose units connected by α 1-4 bonds. The amylopectin

polymer is a highly branched molecule of D-glucose units connected by α 1-4 and α 1-6 linkages.

When starch and water mixtures are heated above certain temperatures the starch granules lose their birefringent properties and swell because of the uptake of water. This series of events is referred to as gelatinization. As gelatinization proceeds in a heated starch-water solution viscosity increases. This is believed to be a direct result of granular swelling (5, 6, 7). Many believe that granular swelling is also responsible for the maximum viscosity developed in a heated starch-water suspension. Miller et. al. (6) has stated that maximum amylograph viscosity is reached after most of the swelling ceases. He proposed that the maximum viscosity is due to an exudate released from the starch granules after swelling occurs. If this theory is correct, the maximum amylograph viscosity of a heated starch-water suspension may result from the gelatinization of the starch and leaching of starch molecules forming a network of highly hydrated amylose and amylopectin starch molecules. It follows from Miller's model that a disruption of the exudate or a reduction in the size of the amylose and amylopectin chains could cause a decrease in the maximum viscosity. Even if the maximum amylograph viscosity of a heated starch-water suspension does not occur as Miller et al. (8) described, but is simply a result of granular swelling, then any partial or total destruction of starch granules could also lead to a reduction in the maximum amylograph viscosity.

Anderson et al. (9, 10) may have observed this breaking of starch chains or disintegration of starch granules in their comparative studies

on roll-and extrusion-cooking of corn and sorghum grits. They observed that under similar cooking conditions, extrusion-cooked corn and sorghum grits had lower amylograph hot paste viscosities than roll-cooked grits. They concluded that the combination of pressure, temperature, and mechanical shear provided by extrusion cooking degraded the starch yielding a cooked viscosity lower than that obtained from the roll cooking process.

Anderson et. al. (9, 10) also observed that the cooked paste viscosity was highest at the lowest extrusion temperatures and decreased as extrusion temperatures increased. Corn and sorghum grits extruded at twenty-five percent moisture were found to give higher amylograph hot paste viscosities than grits extruded at fifteen percent moisture. These results indicate higher extrusion temperatures or lower moistures result in increased thermal degradation of starch and mechanical disintegration of starch granules resulting in lower amylograph viscosities. Two objectives of this study involved the solution viscosities of the wheat starch; A) to compare the viscosities of wheat starch extruded at different operating conditions, and B) to compare the viscosity of extruded starch to those obtained for two commercially available pregelatinized wheat starches.

If extrusion degrads starch more than roll-cooking and possibly other cooking methods, it is reasonable to assume that besides lower viscosity, a higher water solubility of the starch would result from extrusion. Higher starch solubility would be an advantage in processing a nutritious cereal-based drink but a disadvantage to other types of

products, such as gruels. Anderson et. al. (9, 10) have reported that with corn and sorghum grits the water solubilities of the extruded grits were higher than the roll-cooked grits. They observed that extrusion at the combination of higher temperatures and lower moisture resulted in products with higher water solubilities. Moore (11) also observed a trend of increasing water solubles with increasing extrusion temperatures in extruded wheat flours. These two studies indicate that the water solubility of extruded starch may be a function of the extrusion temperature.

It is unlikely that the conditions generated in HT-ST extrusion cooking would degrade starch to the extent found in the preparation of starch dextrins by pyrolysis. However, under certain operating conditions, such as low moisture and high temperature, a pH change may occur during extrusion and cause a glucosidic bond rearrangement in the starch. A reduction in pH has been reported when starch-water suspensions were heated in closed containers, (12, 13, 14). Changes in pH under varying extruder conditions have not been examined. Starch will "brown" as it is cooked at high temperatures. Moore (11) observed as extrusion temperatures increased or screw speed decreased (more residence time within the extruder) color formation increased in the wheat flour extrudate. Color formation would be important if a pregelatinized extruded starch or flour was to be used to make a clear or whitish-opaque gel. It is also unknown whether conditions set up inside an extruder might cause rearrangement of the α 1-4 or α 1-6 bonds forming undigestible linkages. The formation of such linkages would reduce the food-energy value of the starch, and hinder enzymatic digestions for sugar or syrup formation. All three, pH, browning, and odd bond formation were examined in this study.

Starch plays a leading roll in determing a product's texture and density. Many of the HT-ST cooking extruders have been termed "Expanders" because of the tremendous expansion a starch-based formulation can achieve. The word expansion refers to the size or diameter of the extrudate as it compared to the size of the die through which it was extruded. The snack food and breakfast cereal industries have taken advantage of starch expansion to market a wide variety of products with differing shapes, sizes, textures and densities. Williams and Baer (15) have stated that expansion of a starch-based material depends upon A) starch content of the formulation, B) its moisture content, C) the pressure drop across the extruder die, and D) the temperature of extrusion.

The amount of starch in a formulation may directly effect the product expansion. Williams and Baer (15) have found that a one-hundred percent starch formulation will expand five times the diameter of the die opening when extruded at optimized conditions. A formulation of the whole cereal grain which contains sixty-five to seventy-eight percent starch will expand four times the die opening. Some dog food formulas which contain forty to fifty percent starch will expand two to three times the die opening, and defatted oil-seed formulations which contain very little starch will expand one and one-half to three times the die opening. Decreasing the starch content of the formulation will obviously decrease its potential capacity for expansion. Closely related to starch percentage is the source of the starch put into a formulation. Corn starch, wheat starch, acid-modified starch, and rice

flour are considered excellent expanders. Corn meal and corn flour are good expanders, while oat flour and wheat flour are good expanders but require higher extrusion temperatures to obtain good expansion (3).

The moisture content also affects expansion. Inside the extruder the moisture level need only be high enough to gelatinize the starch into a fluid-like mass allowing it to pass through the die. Once outside the extruder the moisture level must be high enough to maintain the fluidity of the starch mass and provide steam to expand the starch, but low enough to allow the starch to harden (gel) and maintain its expanded size. If the moisture content is too high the starch mass will not harden but remain soft and collapse after expansion. Moisture levels of ten to thirty-five percent have been used depending on the type of product to be produced and the amount of expansion desired (16). The pressure differential across the die provides the sudden drop in pressure which allows steam to expand the product. With the pressure differential the expansion of the steam would not be great enough to fully expand the starch. Most products are extruded at 350 to 500 pounds per square inch (17). The pressure within an extruder can, as stated previously, be effected by the design of the extruder barrel and screw, and by varying the size of the die opening. In this work, the die and screw will be kept constant.

The temperatures required for expansion were not discussed by Williams and Baer (16) but they did assume that temperatures high enough for steam formation must be reached. Moore (11) observed that a temperature setting of 150^{o}C gave the greatest expansion with wheat flour.

It was hoped that this study would help explain the effect which varying extruder conditions has upon expansion.

MATERIALS AND METHODS

General

This study was designed to examine the effects of three operating variables of a HT-ST extrusion cooker upon extruded wheat starch. The operating variables included four temperature levels (150, 175, 200, and 225°C), four moisture levels (13, 16, 19, and 22%), and three screw speeds (50, 100, and 150 RPM). That 4 x 4 x 3 design yielded forty-eight sets of conditions. Samples could be obtained for only forty-four sets of conditions. Four samples were unavailable because the combination of the lowest moisture level and the lowest screw speed, continually resulted in failure of the shear pin. The samples not obtained were at 13% moisture, 50 RPM, and 150, 175, 200, and 225°C. (see Table 1, page 45). The coded identification of all the various samples are also given in Table 1. The following five samples were selected to be extruded on three different days, X-002, 005, 024, C34, and 065. The results with those samples were used to examine day to day variations. It was impossible to collect all forty-four samples in one day.

One hundred pounds of wheat starch was donated by the Midwest Solvents Company, Inc., The starch contained 0.1-0.2% protein, and in a water slurry had a pH of 6.76. A Brabender Laboratory Extruder Model 2503 was used in this study. The extruder specifications are as follows:

Barrel Diameter	0.75 inches
Barrel Length	18.75 inches
Barrel Length to Diameter Ratio	25:1
Barrel Volume	47.1 cubic inches
Internal Surface	44.3 square inches
Number of Heated Zones	3
Watts per Zone	1000 watts
Type of Cooling	compressed air cooling
Compression Ratio	5:1
Rod-Shaped Die, Diameter	0.125 inches

The stainless steel screw had a compression ratio of 5:1 and was fitted with a torpedo at the discharge end to fill the large void-volume leading into the die. The barrel was modified with the addition of six triangular grooves 1/8 inch deep running the length of the barrel. The extruder was equipped with a die cap-nozzle (1/8 inch opening) which produced rod-shaped products.

Tempering

The quantity of water needed to temper the wheat starch for a given batch (2270 g) to the desired moisture level was calculated using the following formula.

Water originally in starch + Water added to Starch = Total water after temper

OR

% Starting moisture X 2270 g + Water to be added = Desired Moisture X 2270 g + Water to be added

The wheat starch was placed in a rotating drum (18 inches in diameter, 18 inches long) and water added as a misty spray through an opening at one end of the drum. An extra 15 ml of water was added to each batch to compensate for evaporation and loss. The tempering drum was allowed to run five minutes after the water had been added. The tempered starch was put into a box-blender, blended for five minutes, and placed in tightly fitted cans until use the next day.

Extruder Operating Procedure

1. The extruder was assembled and plugged in.

2. The heating zones were switched on and the temperatures were set for start-up conditions as follows:

Feeder Block	Water Cooled
Zone One	Air Cooled
Zone Two	100°C
Zone Three	135°C

3. When the zones reached temperature and when the die-cap nozzle was very hot to the touch, the extruder was ready for start-up.

4. The screw was turned on and the screw speed adjusted to 100 RPM. The feeder was turned on and adjusted to its slowest speed.

5. In order to prevent plugging on start-up, wheat flour of 25% moisture was slowly fed into the feeder.

6. When the wheat flour started coming through the extruder the zone three temperature (zone closest to die) was adjusted to the desired setting for the first run. The barrel temperatures for the other

two zones remained as set in step 2 throughout the operation for all runs.

7. Tempered wheat starch was introduced when the temperature in zone three reached the desired level.

8. When the wheat starch began exiting through the extruder die the screw speed was adjusted to the desired setting and the feed rate was adjusted to obtain a continuous, uniform outflow.

9. After a uniform outflow and an equilibrium product temperature was established throughout the extruder (approximately 5-10 minutes) the sample was taken. Approximately 200-250 g of each sample was collected and placed in a polyethylene bag for storage.

10. While the sample was being taken the following readings were recorded.

a) Zone three temperature setting

b) Screw speed

c) Feed-rate setting on forced-feeder

d) Product temperature in zone one

e) Product temperature in zone two

f) Product temperature in zone three

Temperature of Product Inside the Barrel

11. When the extruder was switched to a new set of operating conditions it was allowed to come to a new equilibrium before collecting the new sample.

Sample Preparation for Analysis

Approximately 100 g of each sample was ground on an F-No. 4 Quaker City hand grinder. The roughly ground material was then reground on a

small hammer mill with a 0.027 inch screen. The finely ground samples were placed in plastic vials for storage.

Moisture Determination

Moisture determinations were made following the A.O.A.C. Method 14.004 (18).

Expansion

The diameter of the product was used to describe its degree of expansion. Ten random diameter observations were taken using ruled calipers on each sample. The average of ten observations was used as the expansion for that sample. The standard deviation for one sample is 0.0127 inches.

pH

The pH measurements were made on a Beckman Expandomatic SS-2 pH meter. Four grams of ground product was dispersed in 50 ml of distilled water. The pH was taken on this slurry. Two pH determinations were made for each sample after the meter had been standardized with a pH-6.0 buffer. The standard deviation for one sample is 0.07 pH units.

Water Solubility

Five grams of ground sample was added to 180 ml of distilled water in a 250 ml centrifuge bottle. The water was stirred while the sample was added to prevent clumping. The mixture was stirred for thirty minutes with a magnetic stirrer. The stirring bar was removed and washed with 15 ml of distilled water. The washings were allowed to drip into the centrifuge bottle and the contents were stirred with a clean glass stirring rod. The solution was centrifuged for five minutes at 700 g

using an International centrifuge. Duplicate 10 ml aliquots were taken from the top of the supernatant and each aliquot was added to a 100 ml beaker which contained 4.5 g of Celite 545 and which had been previously dried and tared. The beakers were placed in a forced-air oven at 130°C and dried for three hours. After drying, the beakers were placed in a desiccator and allowed to cool before weighing. The percent solubles was calculated using the following equation:

$$\frac{\text{(Beaker weight gain in grams) X 20}}{\text{5 g - (\% Moisture of sample X 5 g)}} = \text{\% Solubles (Dry Basis)}$$

The standard deviation for one sample is $\pm$ 1.3%.

Color

Reflectance color was measured by the Agtron Model -500-A reflectance meter which measures relative spectral reflectance. Two wave lengths were used, 436 nm (blue) and 540 nm (green). Standard discs numbers 56 and 85 were used to standardize the readings in the blue region. Standard discs numbers 71 and 90 were used for the green region. Standardization was checked before taking each sample reading. The standard deviation for one blue color sample is $\pm$ 2.0 units, and the standard deviation for one green color sample is $\pm$ 2.3 units.

Viscosity

Viscosity was measured by a Brookfield Synchro-lectric viscometer using the number one spindle. The calibration of the Brookfield viscometer was checked using standard viscosity solutions provided by the Brookfield Company. The Viscometer was found to be consistent as it

read one centipoise unit low at viscosities between 10 and 100 centipoise. This was considered accurate enough for the relative comparisons to be made. Ten grams of each sample on a dry weight basis was dissolved in 350 ml of 90% Dimethyl Sulfoxide (DMSO) at 55°C (19). After cooling, the solution was made up to 500 ml with 90% DMSO. Readings were made in a 500 ml lipless Berzelius beaker. The readings were converted to centipoise units using the Brookfield conversion chart. The standard deviation for one sample is $\pm$ 0.5 centipoise units.

Enzyme Digestibility

Enzyme digestibility was determined by the method of Shetty, Lineback, and Seib (20). An accurately weighed starch sample (approximately 75 mg) was disolved in approximately 90 ml of 90% DMSO at 55°C in a 100 ml volumetric flask. After five minutes the flask was cooled to 37°C and made to volume with 90% DMSO. Two one milliliter aliquots were placed in test tubes with five milliliters of a glucoamylase solutions (10 I.U. per 1 mg of starch). The tubes were incubated for one hour at 37°C. After one hour ten milliliters of absolute ethanol at 37°C was added to precipitate the enzyme. The tubes were cooled and centrifuged. The clear supernatent was transferred to a clean test tube and equilibrated to 37°C. Two one milliliter aliquots from each of the above test tubes were transferred to two new test tubes and ten milliliters of a glucose oxidase reagent at 37°C were added. After incubating at 37°C for one and one-half hours, four drops of 4N HCl were added to stop the reaction. The absorbance of each tube was measured at 400 nm in a Beckman D. U. Spectrophotometer. A reference sample of raw starch, a glucose standard,

and a blank were run along with each set of samples. The amount of D-glucose released was determined from a standard curve of D-glucose content. The percent starch converted to glucose was calculated correcting for moisture and protein.

Microscopy

Approximately one-half of the ground samples were examined under a polarizing light microscope to determine the percentage of ungelatinized starch granules present. Three wet slides were prepared and examined for each sample. The presence of birefringent zones was used as the criteria for ungalatinized starch.

Scanning electron micrographs were made of selected extruded samples to examine the cellular structure of the extrudate. The samples were mounted on specimen stubs using double-backed tape and were gold plated. The coated specimen were viewed in an ETEC-auto-scanning electron microscope operating at 10-KV accelerating potential. Photographs were taken on Polaroid film (Type P/N).

Statistical Analysis

The inability to obtain four samples created missing data points which made it necessary to utilize a procedure for calculating the analysis of variance where there may be unequal subclasses. A Least Squares Analysis of Variance computer program which can deal will unequal subclass numbers was provided through the Kansas State University Statistical Laboratory. The Least Squares method of calculating the

sample mean and variance is a commonly used method which provides an unbiased estimator with the smallest standard error of any method providing unbiased estimators. The Least Squares method was also advantageous when the program utilized complex statistical methods to estimate unbias least square means for the missing data cells (21).

Because the four missing data cells occurred at the same moisture and screw speed and at all four temperatures, one complete data block was absent for the calculation of moisture-screw speed interactions and three way interactions. For this reason moisture-screw speed interactions and three-way interactions could not be examined.

Significant differences were considered at the five percent probability level. The Least Significant Difference (LSD) test was run for those parameters which were significant. The LSD test provides a numerical value which represents the smallest difference between two sample means that is statistically significant. The program printed out the analysis of variance tables for all the laboratory tests. When a main effect or interaction was significant, the least square means and standard errors were calculated and printed. Whenever missing data was encountered the program calculated by statistical methods the missing least square means (21). This procedure in some instances provided least-squares means not exactly equal to the arithmatic means. Since many of the conclusions in this study are based on this statistical analysis, it is important to note that the differences in the least square means and arithmatic means are generally small and thus do not change the interpretation of the results. An interaction between two treatments indicates that the results obtained from varying one treatment

change as the other treatment is varied. In this study, a moisture-temperature interaction may indicate that a change in a property of the material (viscosity, pH, etc.) obtained by varying temperature may not undergo a proportional change when temperatures are varied at different moisture levels. It may also indicate that the change obtained from varying moisture is different at various temperature levels. This type of dependence makes it necessary when discussing the effects of varying moisture to state the temperature level at which these effects exist. It is possible also, for interactions to be one-sided. Moisture effects may change at different temperatures but temperature effects may be the same at all moisture levels. The opposite may again be true. In very strong interactions, both moisture and temperature may be dependent upon each other and the effect of varying each would depend upon the level of the other.

The interpretation of interactions is not easily accomplished. Graphical representations will be used when discussing interactions and it should be noted that the points in all the graphs are averages over the constant variable(s) (moisture and/or temperature and/or screw speed). When treatments show significant interactions, only trends which are consistent throughout the interaction will be singled out for discussion. Furthermore, the data in the least squares tables with the LSD groupings should be used in conjunction with the graphical depictions of the data to help determine if the trends depicted in the graphs are significant ones.

When examining the statistical results of this study it should be noted that due to the size of this experiment, only one data point was obtained for the majority of extruder conditions. Thus the results of the statistical analysis should only be interpreted as indicating possible trends found within this study. No quantitative relationships should be associated with these trends even though the statistical results are based on and provide quantitative values. For example, as will be seen later, one would conclude from Figure 4, (Page) that viscosity of the extruded starch decreases as temperature increases. This assumption would be a valid one. However in view of obtaining only one sample per set of conditions this observation should be viewed as a trend. Under no circumstances should a conclusion like the following be made: as temperature increases from 175°C to 200°C at 13% moisture the viscosity of the extrudate will decrease five centipoise units.

RESULTS AND DISCUSSIONS

Reproducibility of Results

The reproducibility of the extrusion process was examined by extruding five different starch samples three different days. A one way analysis of variance was run on the data from these samples to obtain an indication of the day to day variability. The data, analysis of variances, and standard deviations are listed in Tables 2-7 (Pages 46-51). The day to day standard deviations were as follows:

Viscosity	± 0.81 centi poise
Expansion	± 0.0028 inches
pH	± 0.13 pH units
Solubility	± 3.6%
Blue Agtron Color	± 6.8 Agtron units
Green Agtron Color	± 7.7 Agtron units

A simple statistical test (the F-test) can help determine if the variance or error which was observed in the reproducibility results is similar to the variance which was found in the original forty-four samples. If these variances are similar it would indicate that if replications had been obtained for all forty-four samples the statistical conclusions would be altered very little. The results of the F-test are listed in Table 8 (Page 52). Four out of the six variances are equal. Only expansion and solubility show different variances and both are just outside of the rejection limits. This does not prove, but indicates that if more replications had been obtained for all the samples the results and conclusions would have varied slightly with the largest differences being in Expansion and Solubility.

Percent Gelatinization

Approximately one-half of the extruded starch samples were examined for ungelatinized starch granules by light-microscopy. None of the samples examined contained a whole starch granule which displayed a maltese cross. Only one sample showed a fraction of a birefringent starch granule. It was concluded that virtually one-hundred percent of the starch in the samples examined had been gelatinized.

Enzyme Digestibility

The digestibility of extruded starch by the enzyme glucoamylase was tested to determine if the digestibility differed from that of the raw uncooked starch. It was theorized that if the digestibility was less, it may have resulted from the formation during extrusion of unnatural nondigestible glucosidic bond linkages. Two extruded starch samples were examined which covered the spectrum of moisture and temperature conditions used in extrusion. The two samples selected were extruded at one extreme of high temperature and low moisture and the other extreme of low temperature and high moisture. The raw starch and the two extruded samples all gave the same digestibility, namely 93%. Since the digestibility of all three samples was exactly the same and since the sample most likely to rearrange had been tested (low moisture, high temperature) it was concluded that bond rearrangement was not important under the conditions used in this work.

Viscosity

Viscosity as measured in this study is a function of the starch polymers' chain lengths, since solution viscosities of the starch were measured in 90% dimethyl sulfoxide. Changes in the size of the amylose and amylopectin chains will cause viscosity differences. An extruded starch which has undergone a high degree of random chain cleavage would have a lower viscosity than one which experienced a lesser degree of degradation. Solution viscosities of the extruded starch samples ranged from 7.5 to 29.2 centipoise units. The data for the individual samplesis given in Appendix I. Viscosities of the extruded starches are considerably lower than the viscosity (86.5 centipoise) of the un-

processed wheat starch and the commercially pregalatinized wheat starches; it is evident that a considerable amount of starch chain-cleavage took place at every set of extruder conditions. It also indicates that extrusion cooking degrades wheat starch more than the two other methods used commercially to produce pregelatinized wheat starch. These findings are similar to those reported by Anderson et. al. (9, 10) where, under similar cooking conditions extrusion cooked corn and sorghum grits had lower amylograph hot paste viscosities than roll-cooked grits.

All three variables, moisture, temperature, and screw speed, showed statistically significant effects upon viscosity. (See Tables 9-11, pages 53-55). A significant interaction between moisture and temperature was also present. In general, lower viscosities were observed at higher temperatures and lower screw speeds whereas moisture displayed no consistent trend in its effect upon viscosity.

The effect of varying screw speed upon starch solution viscosity is a small but a consistent one at all moisture-temperature combinations. As shear rate is increased there is a small increase in viscosity. (See Figure 2, page 69). One might expect that the amount of mechanical shear placed upon the starch within the extruder would effect the viscosity. It would seem possible that the higher screw speed would place more mechanical stress on the starch chains and possibly reduce the chain length and solution viscosity. As Figure 2 illustrates this is not true and there is evidence that the viscosity has not decreased but increased at the higher screw speeds. Even closer examination of

this theory at low moisture and low temperature levels where the starch would be most viscous does not support this concept. The cooking time or retention time within an extruder is however a function of screw speed. If the screw speed is increased the starch is pushed through the extruder at a faster rate decreasing the cooking time. A reduction in cooking time should reduce the amount of thermal starch degradation and thereby increase its solution viscosity. This effect is seen in Figure 2 (Page 69) even though the viscosity change is small. These results indicate that thermal effects are more important to chain cleavage than shearing under these conditions of extrusion.

Moisture showed a significant effect upon viscosity, however, the moisture effects had a significant interaction with the temperature effects. Figure 3 (Page 70) illustrates the effects of moisture upon viscosity. Moving from 13% to 16% moisture the LSD test in Table 11 (Page 55) indicates that there were no significant changes in viscosity. At 19% moisture significant changes do occur at half of the temperature levels. From 19% to 22% moisture there is a significant change in viscosity in all cases but these changes move in opposite directions. It is clear that the effect of moisture upon the solution viscosity of the extruded starch does not show a consistent trend. The effects of varying moisture upon the viscosity of the starch within the extruder and how this effects back pressure and mechanical shear, coupled with the pH changes in the starch makes a complicated set of interrelationships which could not be understood. The information in Figure 3 has been replotted in Figure 4 (Page 71) to more clearly show the effects of temperature upon viscosity. Here increasing temperatures gave decreasing

viscosities for temperatures from 175°C to 225°C. This is a predictable trend for it is logical that higher cooking temperatures would cause greater starch degradation. The failure of this trend to exist between 175°C and 150°C was not explainable. This predominent trend of decreasing viscosity with increasing temperature observed with wheat starch supports the work by Anderson et. al. (9, 10) on corn and sorghum grits. Increasing extrusion temperatures apparently causes greater starch degradation and consequently lower viscosities.

Expansion

Expansion is an important result of extrusion cooking, and represents a measure of the product's diameter. In this study the expansion of the extruded wheat starch ranged from 0.1718 inches (1.37 times the die opening) to 0.4562 inches (3.65 times the die opening) (See Appendix I for all values). Visual observations during sample collection revealed that in some instances the cellular structure hardened almost immediately after the starch left the die. In other instances when the starch expanded, the cellular structure ruptured and the expanded network collapsed to a smaller diameter. This type of behavior indicates the plastic flow properties of the starch matrix were exceeded. Some of the samples displayed this expansion, rupturing of the cellular structure, and collapse. None of the samples reached the five fold expansion factor reported for pure starch by Williams and Baer (15). In general more expansion occurred at the lower moisture levels and the lower temperature levels.

Statistically, moisture and temperature were significant at the 5% level and they displayed a significant two-way interaction (see Tables 12 and 13, pages 56 and 57). Figure 5 (Page 72) shows the effect of moisture upon expansion at the various temperature levels. In all cases the expansion increases as the percent moisture decreases. The effects of temperature interacting with moisture are very prominent at 200°C and 225°C however, these two curves still observe this trend. In general, Figure 5 illustrates that within the range of variables examined in this work, one could expect a higher expansion at a lower moisture level and a lower expansion at a higher moisture level if screw speed and temperature are held constant. This trend has been hearsay for quite a while and no explanation for its existence has been proposed (15, 16). It may however point to an earlier described ideal moisture content which will fluidize and expand the starch, but still be low enough to allow the starch-water mixture to quickly gel after expansion.

The amount of moisture available for steam formation and how it is distributed throughout the starch matrix may help explain the reduced expansion at higher moisture levels. At higher moisture levels there is more moisture available for steam formation and expansion of the starch. This higher moisture content may also reduce the viscosity of the extrudate making it less resistent to expansion by the steam. The lower viscosity and greater capacity for steam formation may combine to form more cells with thinner cell walls producing an extrudate highly

susceptible to the rupturing of cells and collapse of the cellular structure. As the moisture content decreased the number of cells decreased, the cell walls were thicker, and the high viscosity of the extrudate was more resistant to rupture and collapse. Visual observation of the extruded samples shows, in general, the extrudate has more cells and thinner cell walls at higher moisture levels than at lower moistures. (See Figures 22 and 28, Pages 88 and 91). Shrinkage after expansion due to steam condensation creating a slight vacuum within a cell may be another factor contributing to a decrease in expansion. It was obvious during sample collection that at the higher moisture and temperature levels the cellular structure of the product was being ruptured immediately after leaving the die. However, at the higher moisture levels and lower temperatures the extrudate was expanding and shrinkage was occurring. Visual observation could not determine whether this shrinkage was a result of cells rupturing or shrinking due to steam condensation or both. The strong interaction of temperature effects with the moisture effects make these explanations difficult to further clarify or substantiate.

Figure 6 (Page 73) is another representation of the data in Figure 5; Figure 6 shows the effect of varying temperature upon expansion. At first glance this graph portrays an optimum temperature, 175°C, which gives maximum expansion. However, the LSD test in Table 13 (Page ~~80~~ 57) indicates that at all moisture levels the expansion at 150°C and 175°C is not significantly different. This means that 150°C and 175°C yield the same expansion and that statistically nothing can be gained or lost in expansion when switching from one or the other (at a given moisture and screw speed). This fact is more easily seen in Figure

5 where the 150°C and 175°C temperature curves are very much the same. Published extrusion articles (15, 16, 17) have not always included temperature as an important factor in expansion. This may be because many of the expanders in use today rely upon heat dissipation of the mechanical energy used to drive the screw, and the exact extrusion temperatures were never recorded. This study provides three pieces of evidence to support the view that extrusion temperature is an important factor effecting expansion. First is the high statistical significance which the temperature variables displayed and the highly significant interaction between moisture and temperature. Second is the very sharp drop in expansion at higher temperatures as seen in Figures 5 and 6. Third is the fact that these sharp drops in expansion do not occur at every temperature, but occur at certain threshold conditions.

In Figure 6 at 22% moisture and 200°C there was a sharp drop in expansion. At 175°C, the cellular structure is not ruptured and maintains it's expanded state. At 200°C the increased temperature has given the super-heated steam more explosive power which ruptures the starch matrix causing it to collapse. This drop in expansion does not occur at the 16 and 19% moisture levels until 225°C, indicating the importance of moisture and temperature upon the number of cells, their structure and their resistence to expansion. In Figure 5 this higher temperature, 225°C, has already decreased the expansion at 16% moisture. But at 200°C the moisture level must reach 22% before a decrease in expansion occurs. These figures illustrate that a certain combination

of moisture and temperature must be maintained or severe drops in expansion will occur. Temperature is therefore an important factor in expansion, if one wants to avoid rupturing the cell structure and collapse of the starch matrix.

pH

Those extrusion variables which significantly affected pH at the 5% probability level were moisture, temperature and screw speed. The moisture-temperature interaction and temperature-screw speed interactions were also significant (see Tables 14-16, Pages 58 - 60). Interpretation of these results was at first thought to be complicated by the use of alkaline tap water (pH-9.5) to temper the wheat starch. The raw starch had a pH of 6.8 before tempering. After tempering the pH of all samples ranged from 6.9 to 7.0. This is a small increase and should not effect the experiment significantly. The effects of using alkaline tap water in certain instances may not have become evident until after extrusion. The pH of the extruded starch ranged from 5.15 to 7.35. Six samples of the extruded starch had pH values of 7.1 or above (data for all samples is in Appendix I). Three of these six samples occurred at the highest moisture level and five of the six samples occurred at the lowest temperature level. These are the samples one would suspect to have pH values of 7.1 or higher as a result of using alkaline tempering water. How pH values above 7.1 were obtained remains unexplained.

Two commercially pregelatinized wheat starches had pH values of 5.3 and 3.7. The 3.70 pH value is significantly lower than the 5.15-7.35 range of the experimental samples and the 5.30 pH is at the lower end of this range. Information was not available concerning the exact procedures for isolation, gelatinization, and drying of the commercial starches. It is obvious, however, that the final pH of the extruded starches is generally higher than the two commercial starches.

Figure 7 (Page 74) illustrates the effects of varying the moisture content on the pH of the extruded starch. Figure 7 displays no trends except the obvious temperature interaction. The LSD test in Table 15 indicates that many of the points on Figure 7 are not statistically different and that the alike values are intermitent and not consecutive. With the exception of the 150°C and 175°C curves which closely resemble each other, each temperature curve reacts significantly, but oppositely and independently of the others as moisture changes. Figures 8 and 9 (Pages 76 and 77) illustrate the effects of temperature on pH as temperature interacts with both moisture and screw speed respectively. Temperature's interaction with both moisture and screw speed indicates the possibility of a moisture-screw speed interaction. This interaction could not be examined because of missing data. If moisture-screw speed interactions did exist it would complete the triangle for a three-way interaction between moisture, temperature, and screw speed. The existance of a three-way interaction would make interpretation difficult but may help explain the data.

Figures 8 and 9 illustrate temperature's effect on pH as temperature interacts with both moisture and screw speed. Both figures display a trend of decreasing pH with increasing temperature. The decrease in pH was not as large at temperatures of 150°C and 175°C as it was at the other two temperatures. This type of curve is similar to the temperature curves found with viscosity and expansion measurements. It was apparent that as the extrusion temperature increased there was an increasing amount of starch degradation. This increase in degradation was not as severe at 150°C and 175°C as it was at the other two temperatures. Thus, it appears that as starch degradation increased the pH was lowered. The acidic nature of the extruded starch may have been caused by the formation of a few simple organic acids similar to those formed during pyrolysis of starch and under certain conditions of hydrolysis (12, 13, 14). The use of alkaline tap water instead of distilled water to temper the starch may have introduced minerals into the system. These minerals along with the reduction in pH at higher temperatures may accelerate the degradation of the starch. This may explain the lower viscosities observed at higher temperatures.

Figure 10 (Page 78) illustrates the effect of varying the RPM of the extruder screw on the pH of the extruded starch. The LSD test in Table 16 (Page 83) shows that there was no difference between the three screw speeds at 150°C and 175°C. At 200°C and 225°C there was increased pH with increased screw speed. This was a result of shorter cooking times as the screw speed increased. The temperature interaction was prominent and it seemingly splits the graph into two separate trends.

The effects of using alkaline tempering water on the results at the lower temperature levels was not clear. In Figure 10 when the temperature exceeds 175°C it appears to enhance or unmask a predictable trend of increased pH with increased screw speed. This trend was a result of the decrease in residence time of the starch within the extruder barrel as the screw speed was increased.

Water Solubility

The solubility of raw starch was essentially zero. Any solubility of the extruded starch would therefore be a result of the extrusion process. The level of water solubility would be related to the percent gelatinization and the amount of starch degradation occurring during extrusion. The extruded starch samples ranged from 28.5% to 83.5% in water solubility (Data in Appendix I). By varying extrusion conditions, a wide range of solubilities can be obtained. The two commercial pregelatinized wheat starches had solubilities of 9.5% and 13.1%. Those values were much lower than the extruded starch samples.

Moisture, temperature, screw speed, moisture-temperature interaction, and temperature-screw speed interactions were all significant at the 5% level (see Tables 17-19, Pages ~~84-86~~ 61-63). With both interactions significant the evidence points to the presence of a three-way interactions, but it was impossible to test for three-way interactions because of missing data cells.

Figure 11 (Page 79) displays the confusing effect of moisture upon solubility. No trends can be associated with the curves in this graph. The highest solubility occurred at the lowest moisture level,

You have two pages

but high solubilities were also obtained at the highest moisture level. Figures 12 and 13 (Pages 79 and 80) show the effect of increasing temperature upon solubility. Again, no trend was apparent in either figure but higher solubilities were obtained at the highest temperature level. Both Anderson et. al. (7, 8) and Moore (9) have reported experimental evidence that water solubility increases as temperature increases. Moore (9) reported an almost linear relationship while Anderson et. al. (7, 8) reported a curved but direct relationship. Anderson et. al. (7) also reported that the water solubility did not increase as much when temperatures increased from 130°C to 180°C as it did when temperatures rose above 180°C. This tendency for temperature to cause more starch degradation after it exceeded 175°C was not as evident in the solubility curves (Figures 12 and 13) as it was in the viscosity and pH sections (see Figures 4, 8, and 9, pages 71 , 76 , and 77).

At 150°C and 175°C (Figure 14, Page 81) the effects of varying screw speed upon solubility were similar and showed little difference between the three screw speeds. At 200°C and 225°C the slower screw speed (50 RPM) and therefore longer cooking time increased the starch degradation and solubility.

In summary, the water solubility of extruded starch was difficult to predict, even if one knows the temperature, moisture, and screw speed.

Agtron Reflectance Color

The color of the extruded starch samples was measured on an Agtron reflectance meter. The lower the reflectance value the darker the

color. At both the Blue and Green wavelengths the raw uncooked starch displayed a color reading of over 100 which is the highest reading on the scale. The extruded samples ranged from 9.1 to 90.0 in the blue mode and 12.8 to 88.5 in the green mode. The two commercially available pregelatinized wheat starches had Agtron color values of 85 and 90 in the blue mode and 100 plus in the green mode. Thus, almost all of the extruded starch samples underwent more browning during their processing than did the commercial starches. The Analysis of Variance Tables (20 and 21, Pages 64 and 65) show that moisture, temperature, and screw speed had significant effects upon the Agtron color at both wave lengths. There was a significant moisture-temperature interaction in the blue mode. Although moisture was significant in both the blue and green modes the moisture data was confusing. The moisture data in Figure 15 (Page 82) was inconsistent and displayed no trend. Figure 16 (Page 83) appears to show a trend of decreasing color as temp goes from 13 to 19% but the LSD test in Table 23 (Page 67) shows that there was no difference between 13% and 16% moisture. Thus, no trend exists in Figure 16.

Figures 17 and 18 (Pages 84 and 85) display the effects of varying temperature upon the color of the extruded starch. The curves in Figure 17 were inconsistent and displayed no trend. However, more color was apparent at the highest temperature (225°C) than at the lowest (150°C). In Figure 18 the LSD test in Table 23 indicated that there was no difference in the colors at 150°C and 175°C. Thus, figures 17 and 18, only show that the highest extrusion temperature forms more color in the starch extrudate than the lowest temperature. Figures 19 and 20

(Pages 86, 87) are very similar and both show the effect of increasing screw speed upon color. Both graphs display a nearly linear trend of decreasing color with increasing screw speed. Shorter cooking time at higher screw speed reduce the amount of browning in the samples and consequently give them higher Agtron reflectance values.

Morphology of the Rod Shaped Starch Extrudate

Certain extruded starch samples were examined with a scanning electron microscope. The micrographs depict some of the more common features of the extruded starch samples. Figure 21 (Page 88) is a picture of raw starch. The large lens-shaped starch granule encircled in the center of the picture is approximately 20.6 microns in diameter on its flat sides. The smaller starch granule also encircled is approximately 3.2 microns in diameter. These granules are typical of the large and small granules found in wheat starch. Figures 22-27 (Pages 88 - 91) are micrographs at various magnifications of the same sample which was extruded at 19% moisture, 200°C, and 50 RPM. This sample had an average diameter of .3125 (expanded 2.5 times the die opening), a dimethyl sulfoxide-solution viscosity of 19.2 centipoise, water-solubility of 52%, and a pH of 6.2. Figure 22 is a straight-on shot of the end of the rod-shaped product taken at 13X magnification. This picture includes all but a very small percentage of the sample's cross-sectioned area. In Figures 23-25, the magnification was increased and the junction of three cells is seen in more detail. In Figure 24 the cell-wall appears to contain a rib-like structure which results from

uneven expansion of the starch gel as it leaves the die. The cell walls in Figure 24 were 24 microns thick. Figures 26 and 27 are of the same sample and depict the ribbed structure on the inside of a cell and the smoother structure on the outside of the rod-shaped sample.

Figures 28-32 (Pages 91 - 93) are of a sample extruded at 22% moisture, 225°C, and 100 RPM. This sample had an expansion of .1718 inches, a viscosity of 9.0 centipoise, a solubility of 61%, and a pH of 5.48. As the magnification was increased (Figures 28-32) the frail structure of this sample becomes evident. There are many more cells present and the cells are much smaller than in the previous sample. No ribbed cell wall structures were seen in this sample, indicating a homogeneous starch paste and uniform expansion at the die. The cell wall in Figure 31 was approximately one micron thick. This tender sample shatters upon grinding as can be seen in Figure 32.

SUMMARY OF CONCLUSIONS

Three operating variables, moisture, temperature, and screw speed, of an extrusion cooker were examined for their effects upon the following properties of extruded wheat starch: solution viscosity, expansion, pH, water solubility, and color formation. The digestibility, percent gelatinization, and the morphology of the wheat starch extrudate were also examined. One replication was obtained for each set of extrusion conditions. Five samples were selected and extruded three different days to provide an estimate of the day to day variability of the extrusion process. Reproducibility data indicated that the results would still be valid if time would have permitted replication of all the samples.

The microscopy results indicate that virtually 100% starch gelatinization was obtained with extruded wheat starch at each set of conditions examined in this study. The enzyme digestibility data indicated no undigestible glycosidic bonds were formed during extrusion.

The pH's of the extruded starch samples were on the average higher than two commercial gelatinized starches, and lower than the parent starch. More browning or color was also present in extruded starch than in the commercial samples of gelatinized starch.

Extrusion as expected produced a wide variety of samples which expanded to different sizes and textures. Scanning electron micrographs revealed samples which expanded well contained fewer cells of larger diameter and with thicker cell walls. Poor expanding samples in this study contained many cells with very thin, lacy cell walls, which were frequently ruptured.

The conditions used in this study for the extrusion went past simple gelatinization of the starch granules; the amylose and amylopectin chain structures were degraded. This was evident from the lower viscosity and the higher solubility of the extruded samples as compared to the parent starch and the two commercially gelatinized starches.

The three variables, moisture, temperature, and screw speed, effected starch viscosity, pH, water solubility, color, and expansion in different ways. Moisture and temperature were both significant in every test, and there was a strong moisture-temperature interaction present in all but one of the properties. Screw speed was also significant in all but one test. Having a significant effect does not mean a variable will show a consistent, logical, trend in its effect upon a particular analysis. Moisture having been significant in all cases only showed a trend in one of the five analyses. As moisture increased, the diameter of the expanded product decreased. The amount of decrease was related to the temperature as the highly significant moisture-temperature interaction suggested.

Temperature was the most important of the three variables. As temperature increased, viscosity of the starch dissolved in dimethyl sulfoxide decreased, pH decreased, solubility increased, and the amount of color in the samples increased. These changes make it evident that as the extrusion temperature was increased the amount of starch degradation also increased. Temperature and moisture were important factors effecting expansion. The right combination of moisture and temperature must be maintained to optimize expansion.

In the analysis of each starch property it was found that 150°C and 175°C gave similar results which most often were not significantly different. This indicates that after gelatinization occurs there was a temperature buffer zone up to 175°C before starch degradation began to significantly effect the properties of the extruded starch.

Screw speed was significant in all the analyses except expansion. The effects of varying screw speed upon pH were confusing and displayed no trend. As screw speed increased, viscosity increased, at 50 to 100 RPM, solubility decreased at 200°C and 225°C, and the amount of color formed in the product decreased. All of these results indicate that at the highest screw speeds, and therefore the shorter cooking times, lead to less starch degradation and color formation.

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Table 1

Sample Matrix and Identification

TEMPERATURE	Shear Rate	MOISTURE 13%	16%	19%	22%
225°C	150 RPM	X-015	X-034	X-054	X-074
	100 RPM	X-014	X-033	X-053	X-073
	50 RPM	N. A.	X-032	X-052	X-072
200°C	150 RPM	X-001	X-020	X-040	X-060
	100 RPM	X-002	X-021	X-041	X-061
	50 RPM	N. A.	X-022	X-042	X-062
175°C	150 RPM	X-004	X-023	X-043	X-063
	100 RPM	X-005	X-024	X-044	X-064
	50 RPM	N. A.	X-025	X-045	X-065
150°C	150 RPM	X-007	X-026	X-046	X-066
	100 RPM	X-008	X-027	X-047	X-067
	50 RPM	N. A.	X-028	X-048	X-068

X - Sample I. D. No.

X-000 - Raw Starch

N. A. - Not Available

Table 2

Data, Analysis of Variance, and Standard Deviations

For Day to Day Changes in Viscosity

	Data in Centipoise Units				
Replication	X-002[a]	X-005	X-024	X-034	X-065
1	19.6	23.0	26.7	11.6	27.2
2	21.9	18.5	24.4	14.5	26.6
3	21.6	19.6	25.5	14.3	26.1

Analysis of Variance

Source	Degrees of Freedom	Sum of Squares	Mean Squares
Between Samples	4	325.8	81.40
Error Within Samples	10	22.6	2.26
Total	14	348.4	

Day to day standard deviation for viscosity = $\pm$ 0.81 centipoise units.

[a]Coded sample identification, see Table 1, page 45.

Table 3

Data, Analysis of Variance, and Standard Deviation

For Day to Day Changes in Expansion

	Data in Inches				
Replication	X-002[a]	X-005	X-024	X-034	X-065
1	.4250	.4250	.4031	.2594	.3156
2	.4187	.4282	.3812	.2781	.3000
3	.4031	.4313	.3937	.3162	.3125

Analysis of Variance

Source	Degree of Freedom	Sum of Squares	Mean Squares
Between Samples	4	.05089	.01267
Error Within Samples	10	.00231	.00023
Total	14	.05300	

Day to day standard deviation for expansion = $\pm$ .00277 inches.

[a]Coded sample identification, see Table 1, page 45 .

Table 4

Data, Analysis of Variance, and Standard Deviations

For Day to Day Changes in pH

	Data in pH units				
Replication	X-002[a]	X-005	X-024	X-034	X-065
1	6.55	6.84	7.05	5.52	6.92
2	6.70	6.84	7.17	6.14	7.00
3	6.84	6.99	7.09	6.44	6.86

Analysis of Variance

Source	Degree of Freedom	Sum of Squares	Mean Squares
Between Samples	4	2.07	.517
Error Within Samples	10	.53	.053
Total	14	2.60	

Day to day standard deviation for pH = $\pm$ 0.13 pH units.

[a]Coded sample identification, see Table 1, page 45 .

Table 5

Data, Analysis of Variance, and Standard Deviations

For Day to Day Changes in Solubility

	Data in Percent				
Replication	X-002[a]	X-005	X-024	X-034	X-065
1	73.5	47.7	48.2	46.8	33.6
2	74.4	69.4	58.0	45.7	45.5
3	70.2	63.0	55.5	49.4	39.9

Analysis of Variance

Source	Degrees of Freedom	Sum of Squares	Mean Squares
Between Samples	4	424.4	106.1
Error Within Samples	10	388.3	38.8
Total	14	812.7	

Day to day standard deviation for solubility = $\pm$ 3.6%.

[a]Coded sample identification, see Table 1, page 45 .

Table 6

Data, Analysis of Variance, and Standard Deviations

For Day to Day Changes in Color Blue

	Data in Agtron Units				
Replication	X-002[a]	X-005	X-024	X-034	X-065
1	31.0	55.2	48.8	44.8	46.0
2	47.8	59.5	72.5	69.0	76.0
3	45.0	59.5	65.8	55.0	45.5

Analysis of Variance

Source	Degree of Freedom	Sum of Squares	Mean Squares
Between Samples	4	797.7	199.6
Error Within Samples	10	1367.1	136.7
Total	14	2164.8	

Day to day standard deviation for color blue = $\pm$ 6.75 Agtron units.

[a]Coded sample identification, see Table 1, page 45.

Table 7

Data, Analysis of Variance, and Standard Deviations

For Day to Day Changes in Color Green

	Data in Agtron Units				
Replication	X-002[a]	X-005	X-024	X-034	X-065
1	35.8	52.0	54.9	52.4	44.0
2	57.8	61.5	73.0	77.5	77.0
3	42.0	55.5	65.2	58.0	36.0

Analysis of Variance

Source	Degrees of Freedom	Sum of Squares	Mean Squares
Between Samples	4	708054.5	177013.6
Error Within Samples	10	1760.3	176.0
Total	14	709814.8	

Day to day standard deviation for color green = $\pm$ 7.7 Agtron units.

[a]Coded sample identification, see Table 1, page 45.

Table 8

F-Test for Equal Variances

	Viscosity	Expansion	pH	Solubility	Color: Blue	Color: Green
Mean Square Error For Reproducibility Data	2.26	0.00023	.053	38.8	136.7	176.0
Mean Square Error For Original Data	2.36	0.0007	.1354	10.6	85.9	140.4
F-Value	1.04	3.04	2.60	3.59	1.59	1.25
F-Value for Rejection *	2.77	2.77	2.77	2.77	2.77	2.77
Conclusion	$S_1^2 = S_2^2$	$S_1^2 \; S_2^2$	$S_1^2 = S_2^2$	$S_1^2 \; S_2^2$	$S_1^2 = S_2^2$	$S_1^2 = S_2^2$

* If the Calculated F Value is less than the rejection F-Value then the variances are equal.

Table 9

Analysis of Variance for Viscosity

Source	Degrees of Freedom	Sums of Squares	Mean Squares	F-Ratio	Probability
Moisture	3	49.34	16.44	6.96	0.0022 *
Temperature	3	960.59	320.20	135.55	0.0000 *
RPM	2	17.30	8.65	3.66	0.0441 *
Moist. x Temp.	9	198.29	22.03	9.33	0.0000 *
Temp. x RPM	6	26.82	4.47	1.89	0.1320
Residual	20	47.24	2.36		
Total	43	1373.06			

* Significant at 5% probability level.

Table 10

Least-Square Means of Viscosity at Different Screw Speeds

	50	100	150
Viscosity	17.2	18.4[a]	18.8[a]

[a]The LSD test indicates these values are not significantly different.

LSD Value = 0.95 centipoise

Table 11

Least Square Means for the Viscosity Moisture-Temperature Interaction With LSD Groupings Labeled

		Moisture Levels			
		13%	16%	19%	22%
Temperature Levels	225°C	11.91^{a}	10.63^{a}	10.79^{a}	8.66
	200°C	19.10^{a}_{x}	19.86^{a}	$18.43^{a}_{x,y}$	13.16
	175°C	23.83^{a}	24.79^{a}	19.89_{y}	24.09^{a}_{x}
	150°C	20.52^{a}_{x}	21.76^{a}	17.09_{x}	25.59_{x}

[a]The moistures with their means sharing the same superscript at a given temperature level are not significantly different.

[x,y]The temperatures with their means sharing the same subscript at a given moisture level are not significantly different.

LSD Values = 1.8 Centipoises

Table 12

Analysis of Variance for Expansion

Source	Degrees of Freedom	Sums of Squares	Mean Squares	F-Ratio	Probability
Moisture	3	0.0879	0.0293	44.764	0.0000 *
Temperature	3	0.1577	0.525	80.301	0.0000 *
RPM	2	0.0042	0.0021	3.252	0.0599 *
Moist. X Temp.	9	0.0242	0.0027	4.122	0.0040 *
Temp. X RPM	6	0.0036	0.0006	0.923	0.4995
Residual	20	0.0131	0.0007		
Total	43	0.3231			

* Significant at 5% probability level.

Table 13

Least Square Means for the Expansion Moisture-Temperature Interaction with LSD Groupings Labeled

		Moisture Levels			
		13%	16%	19%	22%
Temperature Levels	225°C	.31268	$.21770^{a}$	$.20523^{a}$	$.18853^{a}_{y}$
	200°C	$.40587_{x}$	$.36666^{a}$	$.35313^{a}$	$.20103_{y}$
	175°C	$.42568^{a}_{x}$	$.40729^{a,b}_{x}$	$.38439^{b}_{x}$	$.33229_{x}$
	150°C	$.41229^{a}_{x}$	$.39896^{a}_{x}$	$.38329^{a}_{x}$	$.31563_{x}$

^{a,b}The moistures with their means sharing the same superscript at a given temperature level are not significantly different.

^{x}The temperatures with their means sharing the same subscript at a given moisture level are not significantly different.

LSD Values = 0.0299 Inches

Table 14

Analysis of Variance for pH

Source	Degrees of Freedom	Sums of Squares	Mean Squares	F-Ratio	Probability
Moisture	3	0.2795	0.0932	6.883	0.0023 *
Temperature	3	12.5868	4.1956	309.916	0.0000 *
RPM	2	0.4265	0.2132	15.753	0.0001 *
Moist. X Temp.	9	1.5836	0.1760	12.997	0.0000 *
Temp. X RPM	6	0.2333	0.0389	2.872	0.0348 *
Residual	20	0.2708	0.1354		
Total	43	17.7812			

* Significant at 5% probability level.

Table 15

Least-Square Means for the pH Moisture-Temperature Interaction with LSD Groupings Labeled

		Moisture Levels			
		13%	16%	19%	22%
Temperature Levels	225°C	6.243	5.330^{a}	5.590	5.416^{a}
	200°C	6.558^{a}	6.496^{a}	6.270	6.120
	175°C	$6.835^{a,b}_{x}$	7.093_{x}	6.720^{a}	6.946^{b}
	150°C	6.835_{x}	7.170^{a}_{x}	6.980	7.260^{a}

^{a,b}The moistures with their means sharing the same superscript at a given temperature level are not significantly different.

^{x}The temperatures with their means sharing the same subscript at a given moisture level are not significantly different.

LSD Value = 0.14 pH Units.

Table 16

Least Square Means for the pH Temperature-Shear Rate Interaction With LSD Groupings Labeled

		Shear Rates (RPM) 150	100	50
Temperature Levels	225°C	5.832	5.690	5.4129
	200°C	6.565	6.400	6.119
	175°C	6.980	6.877	6.838
	150°C	7.100	7.002	7.081

[a,b]The shear rates with their means sharing the same superscript at a given temperature level are not significantly different.

LSD Value = 0.12 pH units

Table 17

Analysis of Variance for Solubility

Source	Degrees of Freedom	Sums of Squares	Mean Squares	F-Ratio	Probability
Moisture	3	1977.567	659.189	62.397	0.0000 *
Temperature	3	2743.712	914.570	86.571	0.0000 *
RPM	2	582.313	291.156	27.560	0.0000 *
Moist. X Temp.	9	2088.316	232.035	21.964	0.0000 *
Temp. X RPM	6	598.772	99.795	9.446	0.0000 *
Residual	20	211.288	10.564		
Total	43	7071.047			

* Significant at 5% probability level.

Table 18

Least Square Means for the Solubility Moisture-Temperature Interaction with LSD Groupings Labeled

		Moisture Levels			
		13%	16%	19%	22%
Temperature Levels	225°C	87.66	56.66	49.19	65.61
	220°C	70.31	58.26	40.95	40.02
	175°C	48.24	47.78	48.98	33.03
	150°C	50.26	43.28	49.13	37.78

[a]The moistures with their means sharing the same superscript at a given temperature level are not significantly different.

[x]The temperatures with their means sharing the same subscript at a given moisture level are not significantly different.

LSD Value = 3.80%

Table 19

Least Square Means for the Solubility Temperature-Shear Rate Interaction with LSD Groupings Labeled

		Shear Rate (RPM)		
		150	100	50
Temperature Levels	225°C	59.41[a]	60.18[a]	74.75
	200°C	42.00[x]	52.32	62.83
	175°C	43.36[a,x]	45.85[a,x]	44.31[a,x]
	150°C	43.87[a,x]	46.59[a,x]	44.87[a,x]

[a]The shear rates with their means sharing the same superscript at a given temperature level are not significantly different.

[x]The temperatures with their means sharing the same subscript at a given shear rate are not significantly different.

LSD Value = 3.29%

Table 20

Analysis of Variance for Blue Agtron Color

Source	Degrees of Freedom	Sums of Squares	Mean Squares	F-Ratio	Probability
Moisture	3	2032.32	677.44	7.884	0.0012 *
Temperature	3	5425.80	1808.60	21.048	0.0000 *
RPM	2	2648.80	1324.40	15.413	0.0001 *
Moist. X Temp.	9	1903.31	211.50	2.461	0.0449 *
Temp. X RPM	6	393.47	65.58	0.763	0.6072
Residual	20	1718.52	85.93		
Total	43	13643.23			

* Significant at 5% probability level.

Table 21

Analysis of Variance for Green Agtron Color

Source	Degrees of Freedom	Sums of Squares	Mean Squares	F-Ratio	Probability
Moisture	3	1919.48	639.83	4.556	0.0137 *
Temperature	3	1495.35	498.45	3.549	0.0330 *
RPM	2	3267.99	1634.00	11.636	0.0004 *
Moist. X Temp.	9	2005.52	222.84	1.587	0.1863
Temp. X RPM	6	964.64	160.77	1.145	0.3734
Residual	20	2808.58	140.43		
Total	43	11614.70			

* Significant at 5% probability level.

Table 22

Least Squares Means for the Blue Agtron Color for Screw Speed

	Screw Speed		
	50 RPM	100 RPM	150 RPM
Blue Agtron Color	44.47	54.01	64.57

LSD Value = 4.69 Agtron Color Units

Table 23

Least Square Means for the Blue Agtron Color Moisture-Temperature Interaction with LSD Groupings Labeled

		Moisture Levels			
		13%	16%	19%	22%
Temperature Levels	225°C	29.43_{x}	42.36^{a}	49.90^{a}_{x}	42.94^{a}
	200°C	35.74^{a}_{x}	26.83^{a}	60.00^{b}_{x}	$61.60^{b}_{x,y}$
	175°C	$58.18^{a,b}_{y}$	54.76^{a}	71.73^{b}_{y}	55.00^{a}_{x}
	150°C	60.48^{a}_{y}	74.60^{b}	75.33^{b}_{y}	$70.66^{a,b}_{y}$

^{a,b}The moistures with their means sharing the same superscript at a given temperature level are not significantly different.

^{x,y}The temperatures with their means sharing the same subscript at a given moisture level are not significantly different.

LSD Value = 10.8 Agtron Color Units

Table 24

Least Square Means for the Green Agtron Color for Moisture, Temperature and Shear-Rate with LSD Groupings Underlined

Moisture	13%	16%	19%	22%
Green Agtron Reading	51.02[a]	55.86[a,b]	69.47	62.50[b]
Temperature	225°C	200°C	175°C	150°C
Green Agtron Reading	53.42[a]	54.66[a]	62.55[b]	68.22[b]
Shear Rate	50 RPM	100 RPM	150 RPM	
Green Agtron Reading	48.69	59.41	71.04	

[a,b] The LSD test indicates that the underlined values are not significantly different.

Moisture LSD Value = 6.92 Agtron Color Units

Temperature LSD Value = 7.47 Agtron Color Units

Screw Speed LSD Value = 5.99 Agtron Color Units

Figure 2

Viscosity VS Screw Speed

(Averaged Over All Moistures & Temperatures)

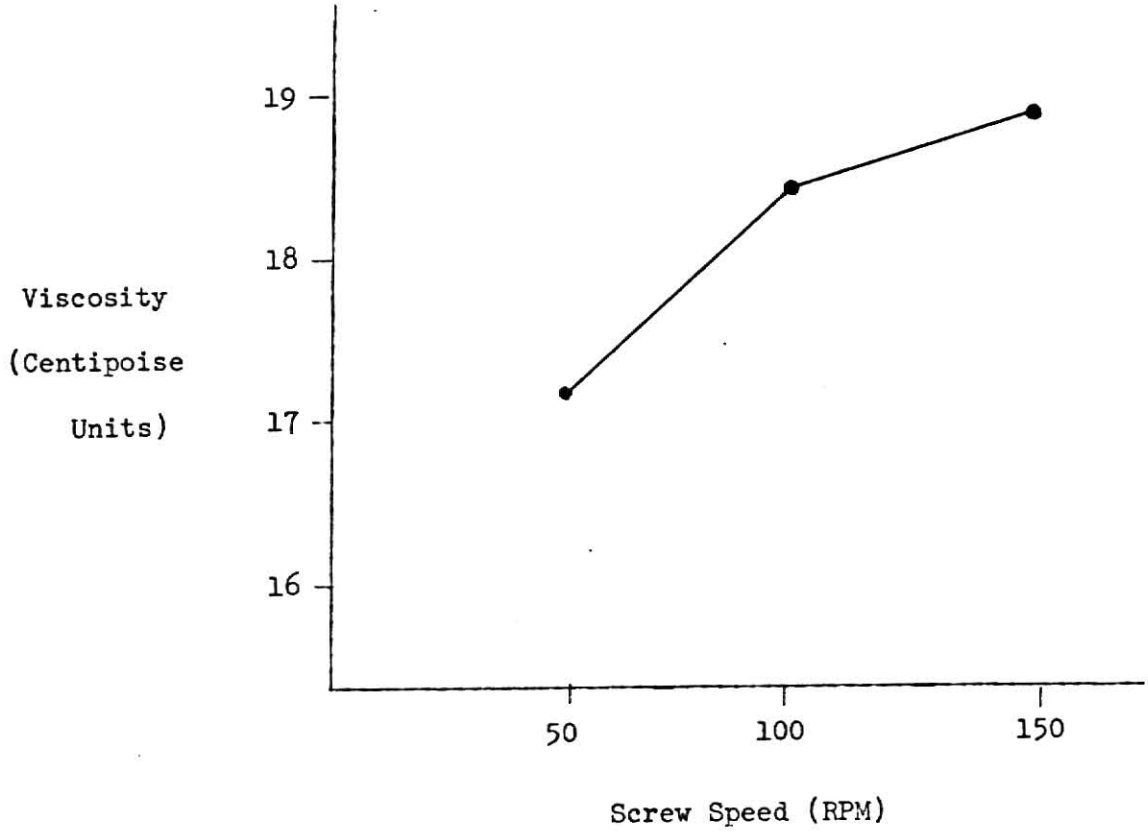


Figure 3

Viscosity VS Moisture

(Averaged Over All Screw Speed)

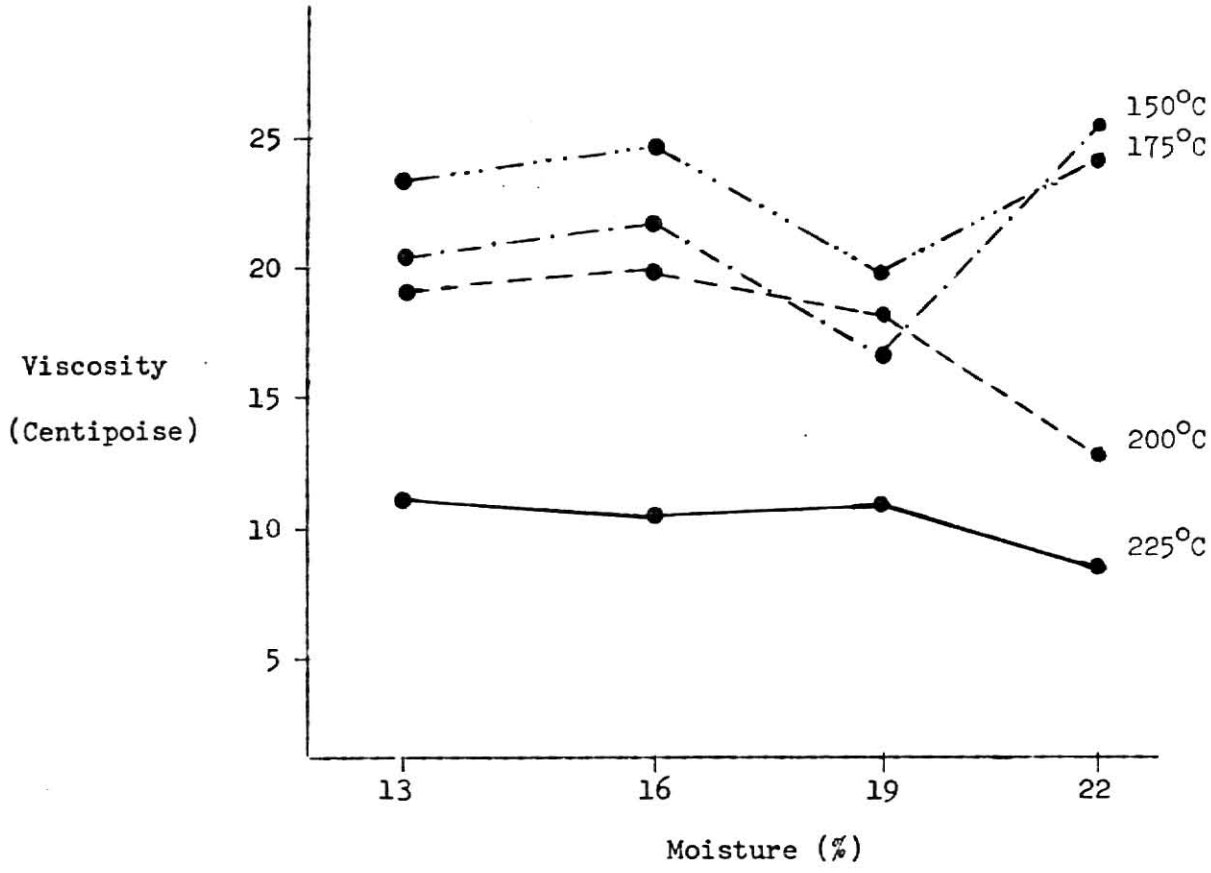


Figure 4

Viscosity VS Temperature

(Averaged Over All Screw Speeds)

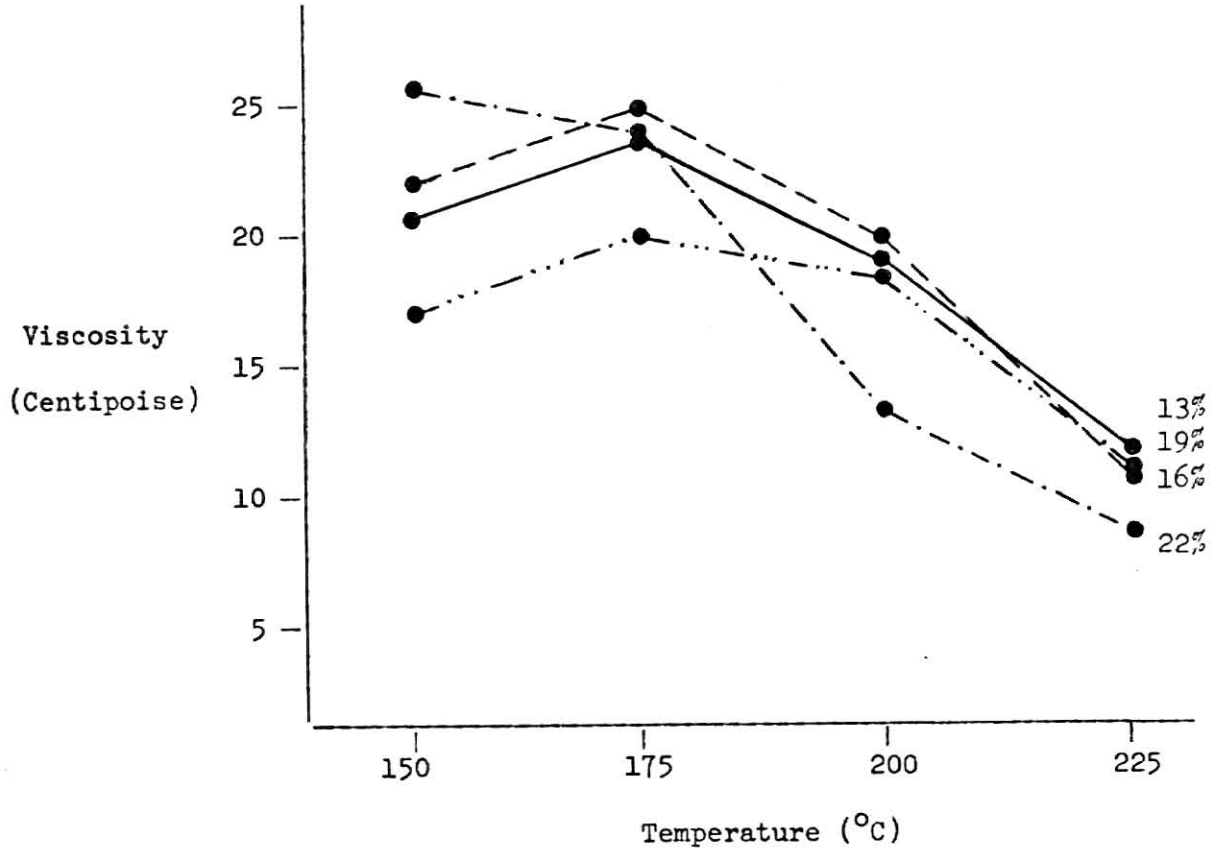
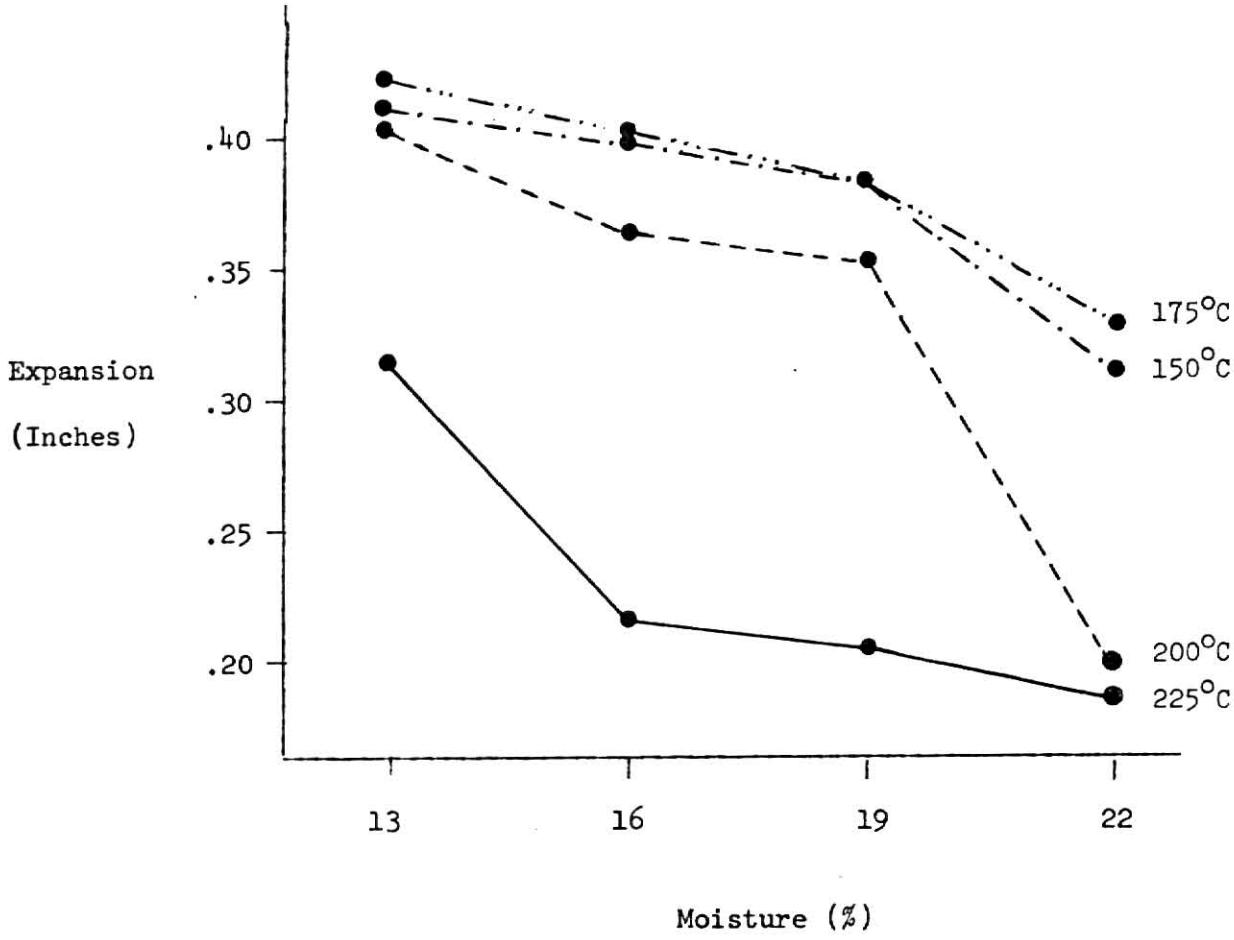


Figure 5

Expansion VS Moisture

(Averaged Over All Screw Speeds)

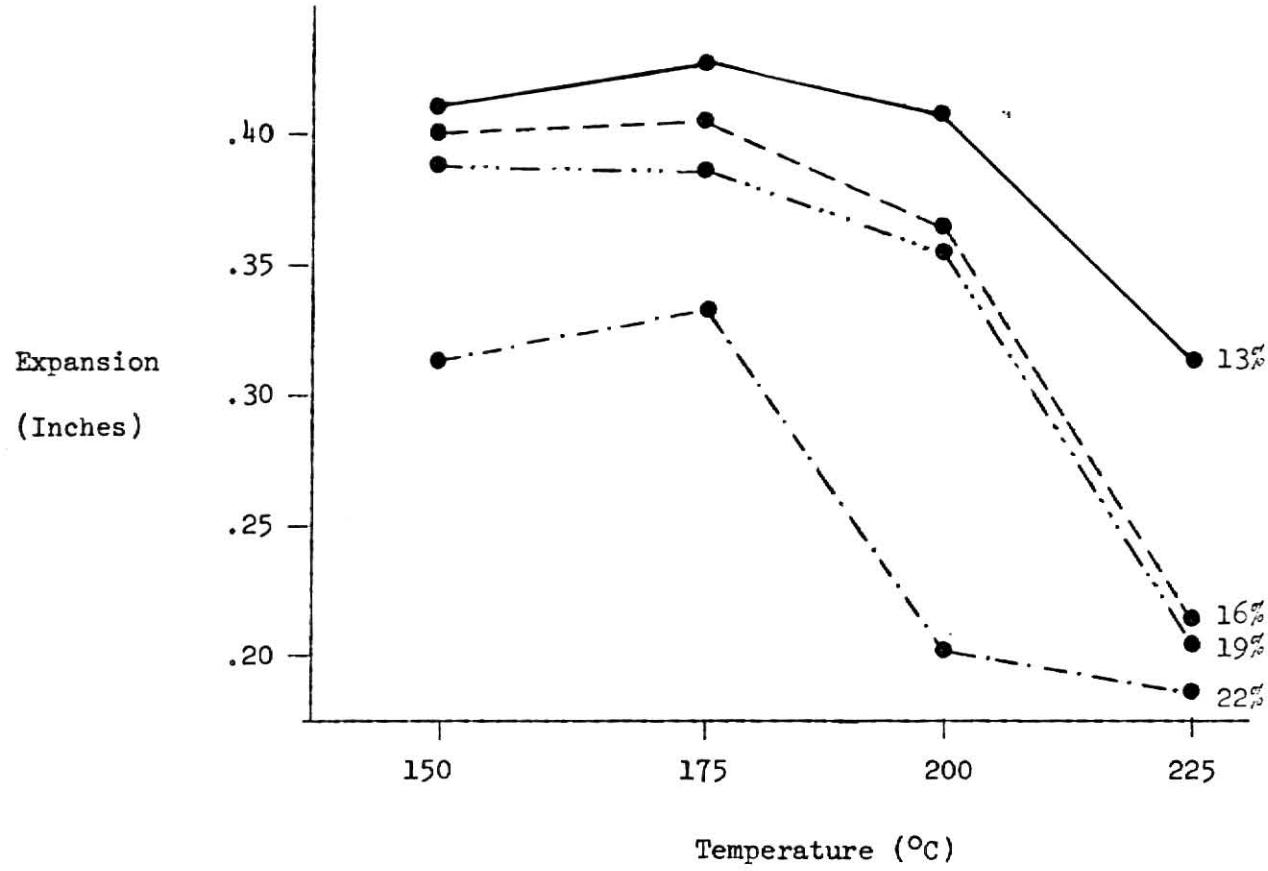


Die Opening = 0.125 Inches

Figure 6

Expansion VS Temperature

(Averaged Over All Screw Speeds)



Die Opening = 0.125 Inches

Figure 7

pH VS Moisture

(Averaged Over All Screw Speeds)

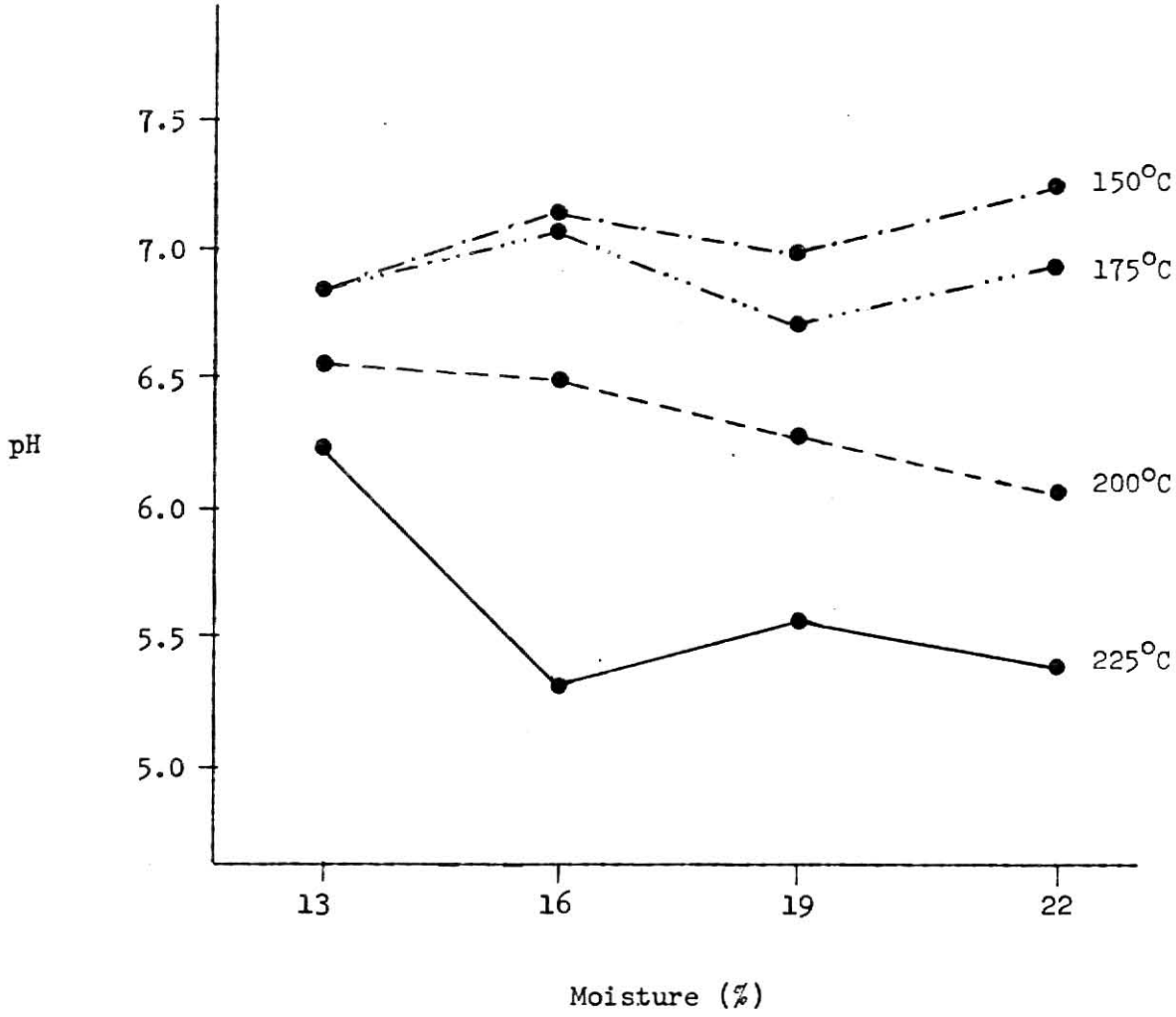


Figure 8

pH VS Temperature

(Averaged Over All Screw Speeds)

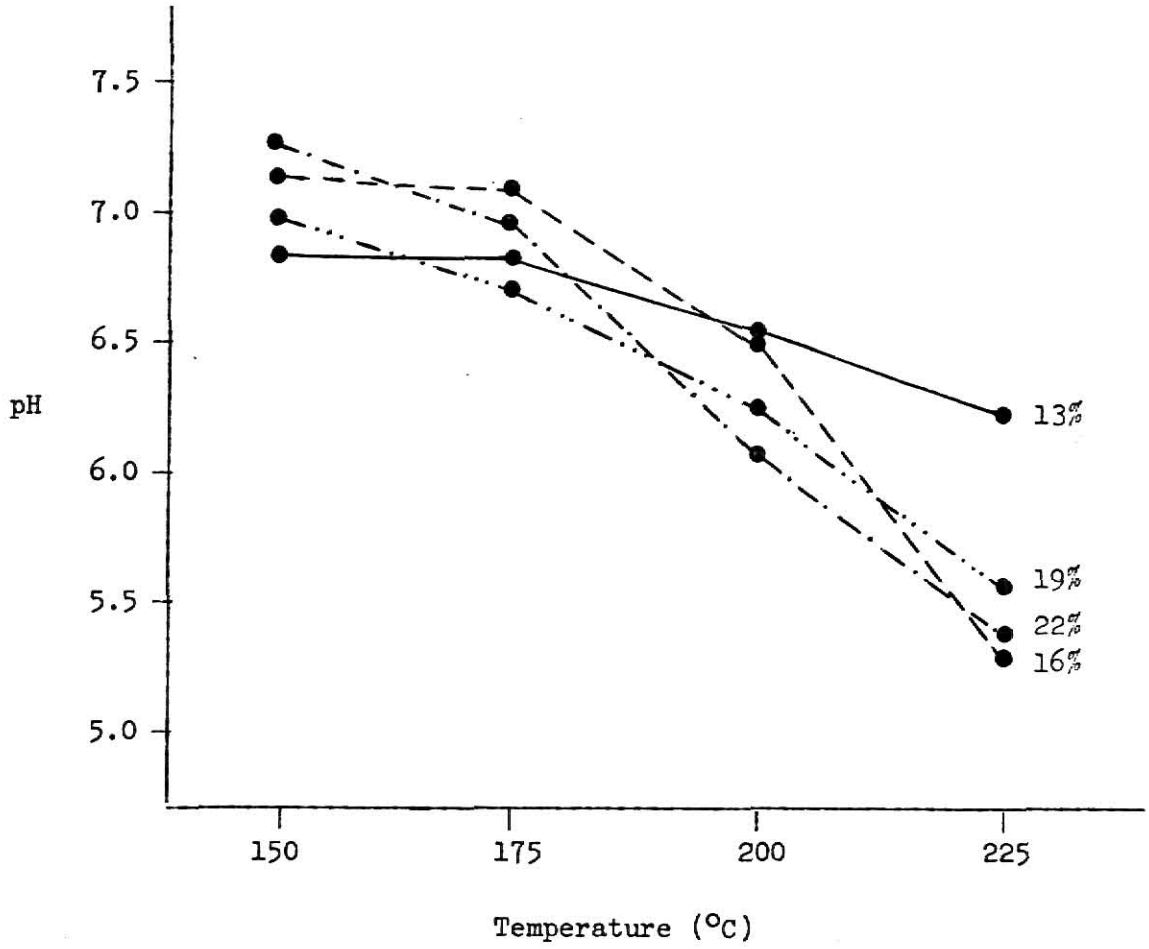


Figure 9

pH VS Temperature

(Averaged Over All Moisture Levels)

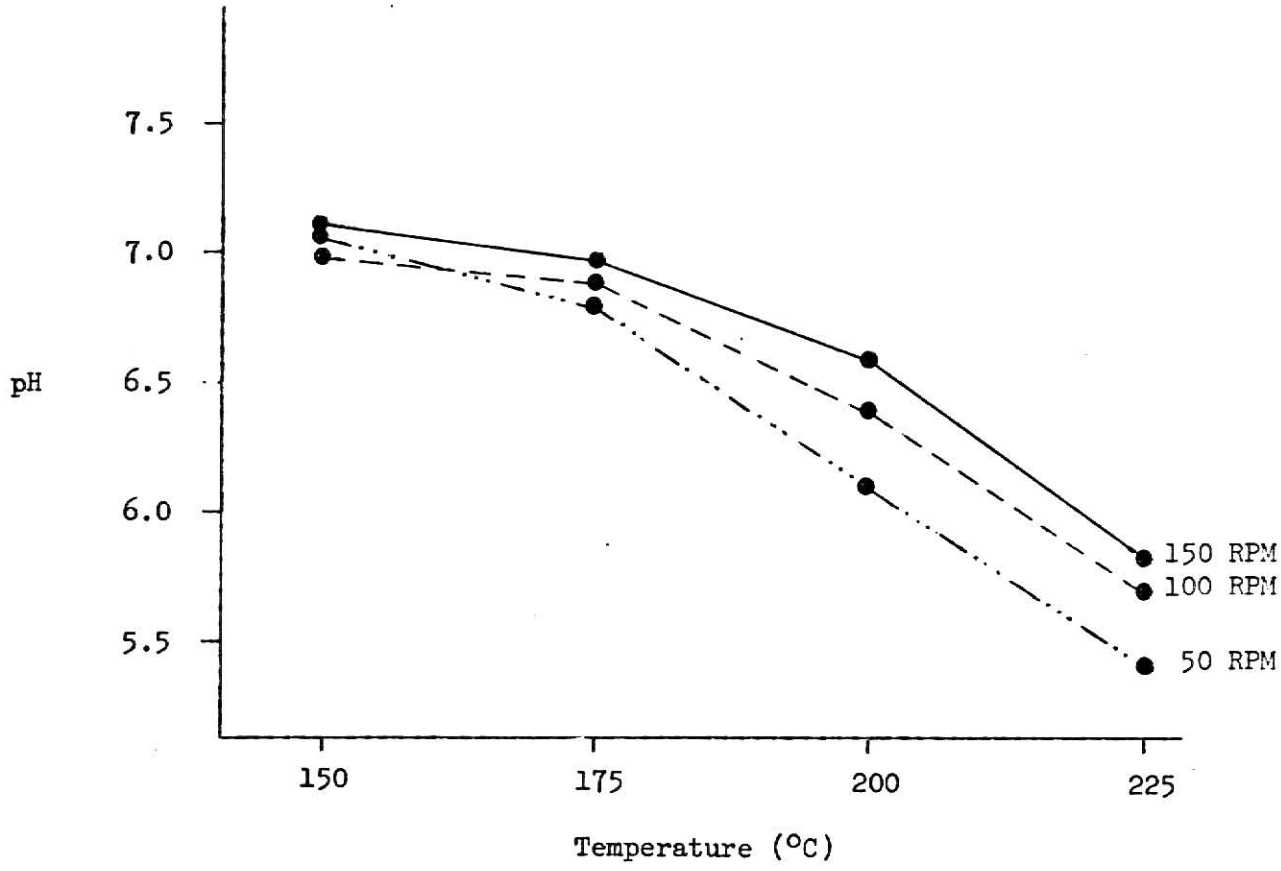
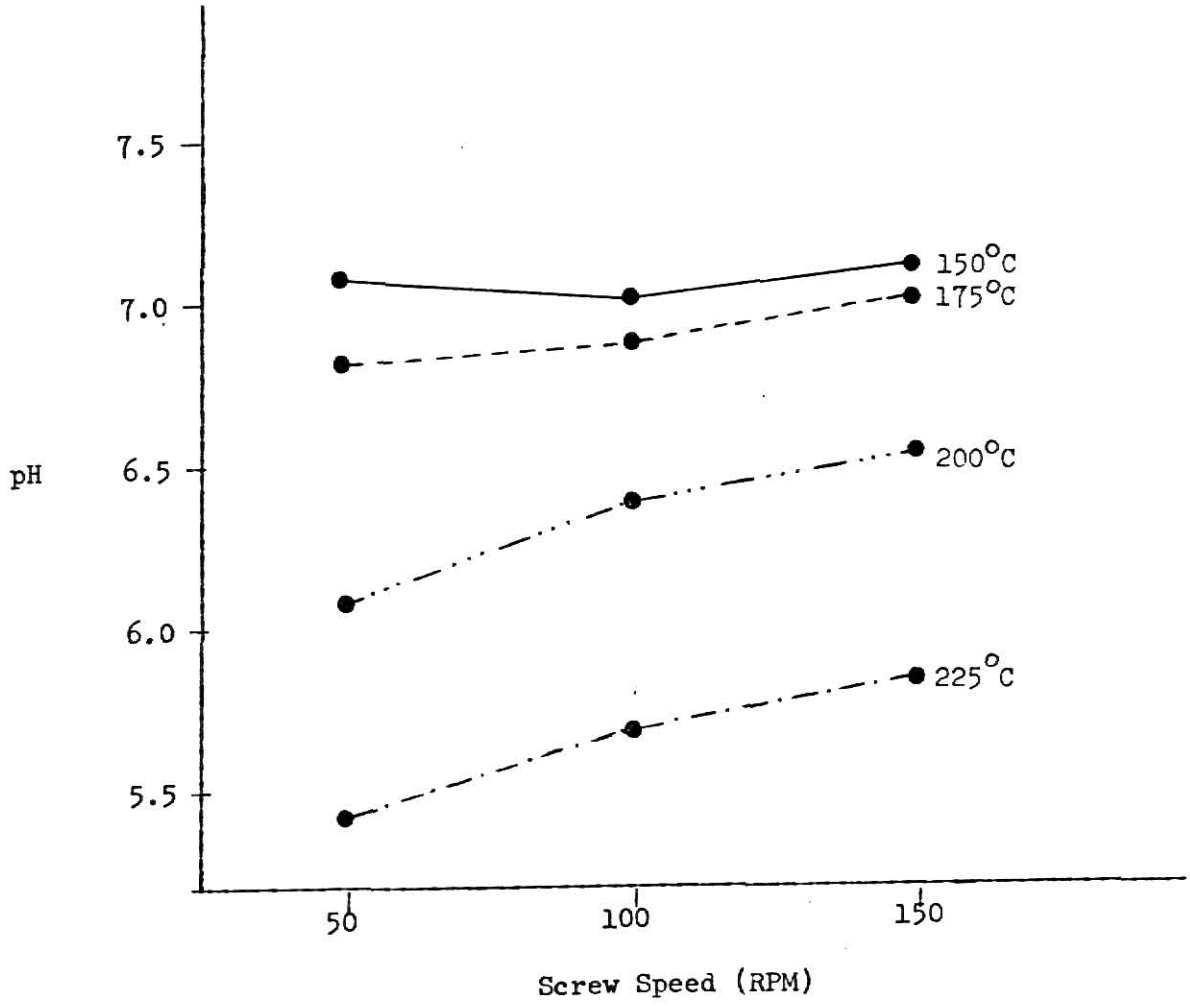


Figure 10

pH VS Shear Rate

(Averaged Over All Moisture Levels)



THIS BOOK WAS BOUND WITH TWO PAGES NUMBERED 79. THESE PAGES ARE DIFFERENT.

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Figure 11

Water Solubility VS Moisture

(Averaged Over All Screw Speeds)

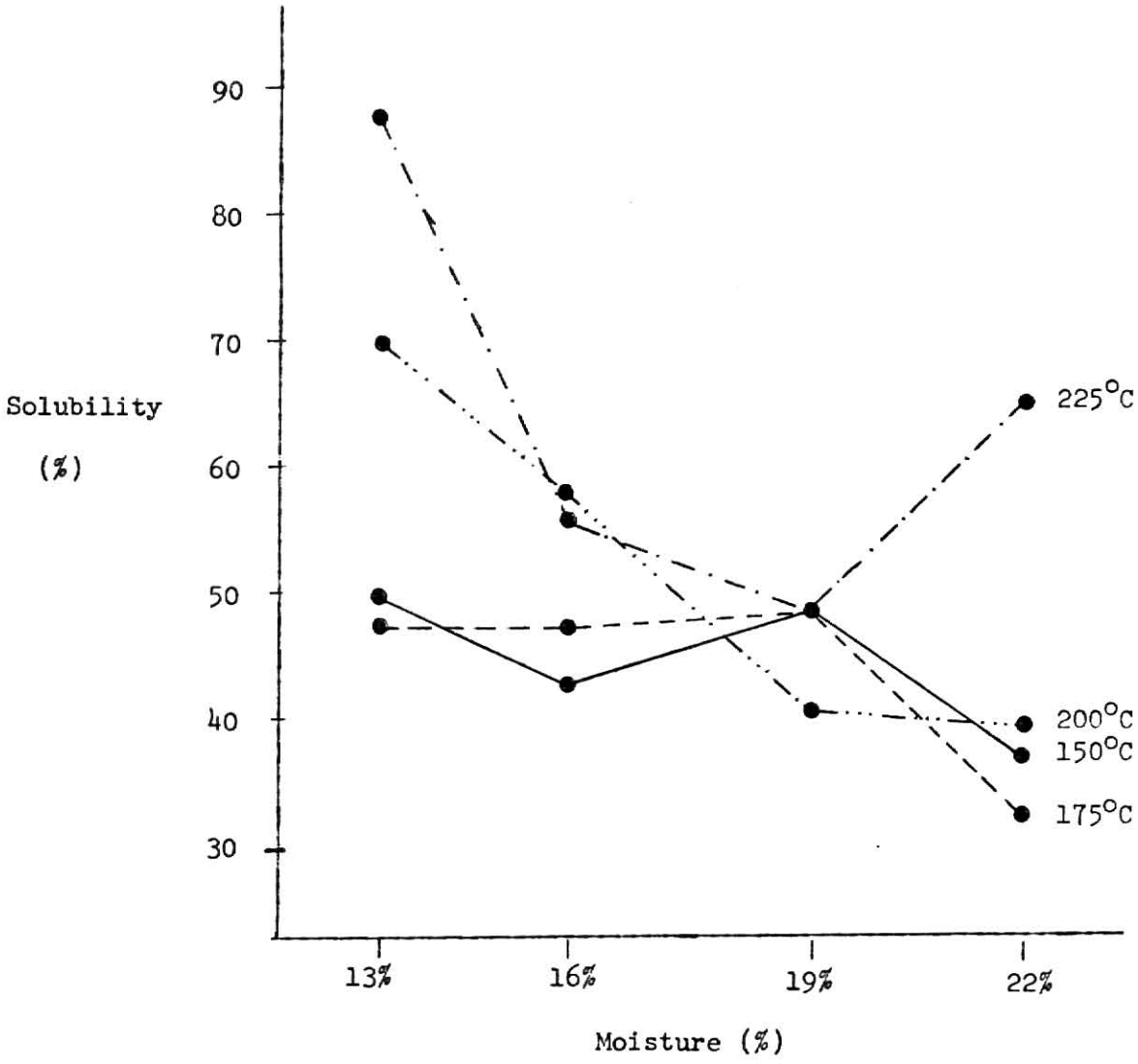


Figure 12

Water Solubility VS Temperature

(Averaged Over All Screw Speeds)

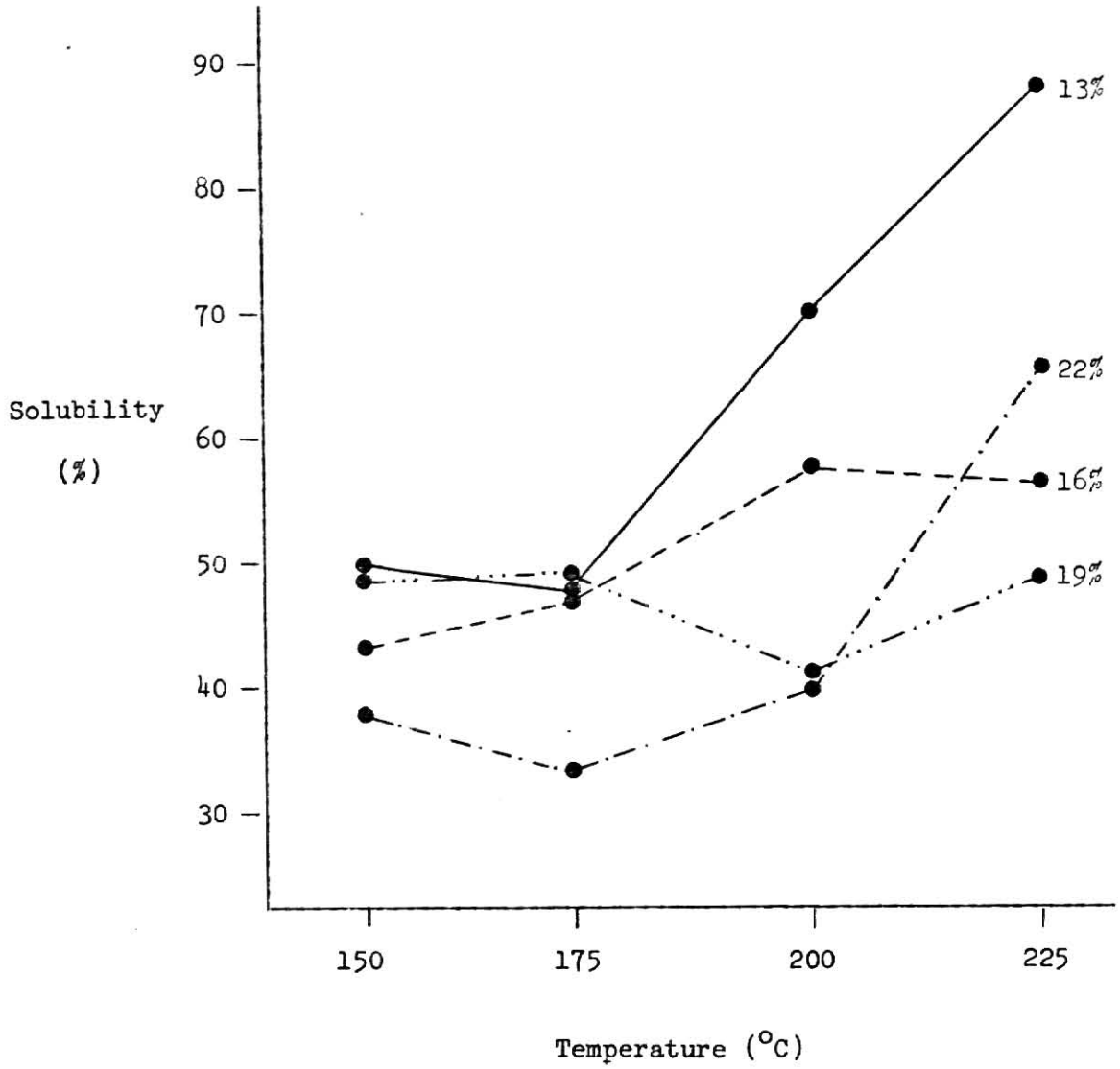


Figure 13

Water Solubility VS Temperature

(Averaged Over All Moisture Levels)

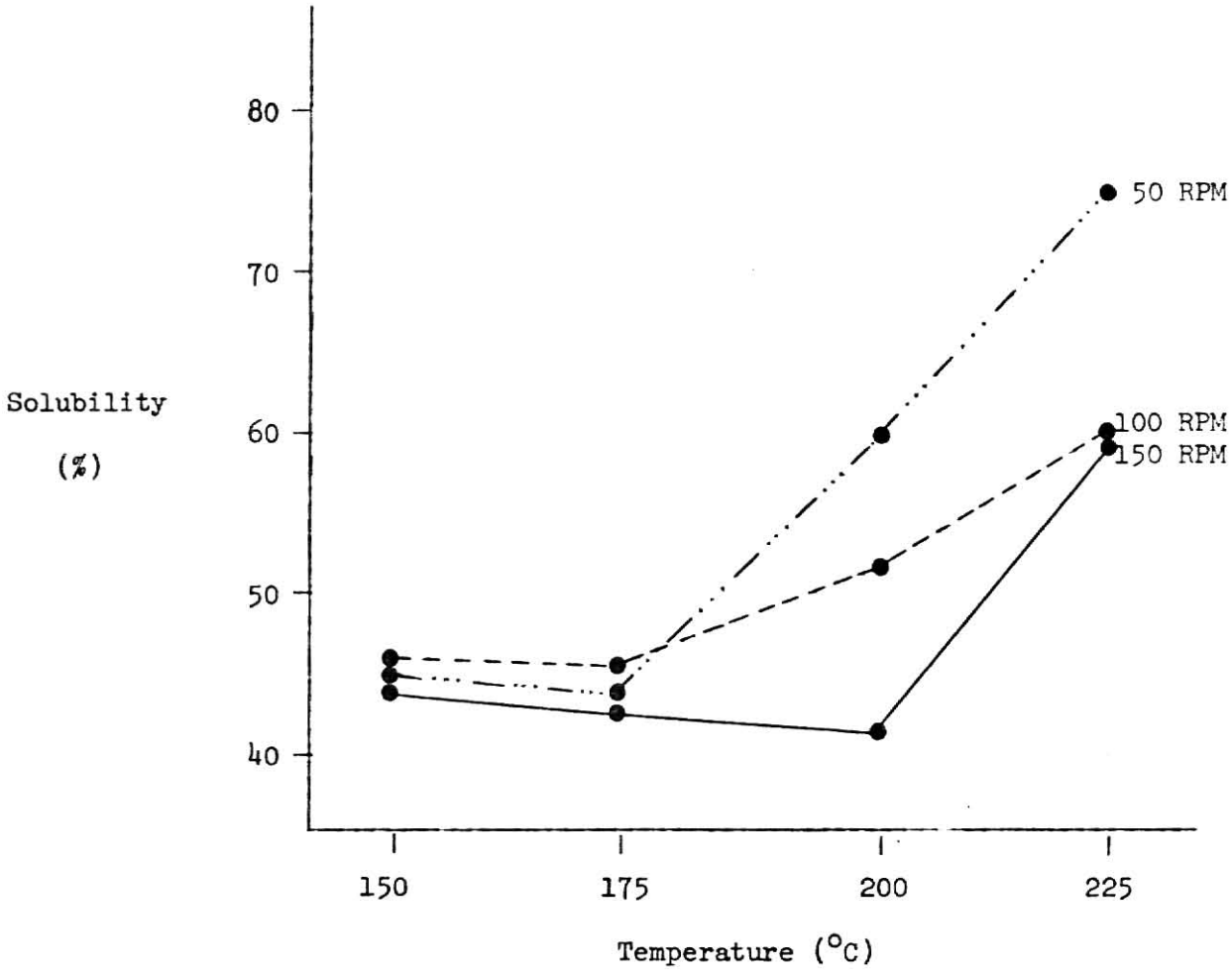


Figure 14

Water Solubility VS Shear Rate

(Averaged Over All Moisture Levels)

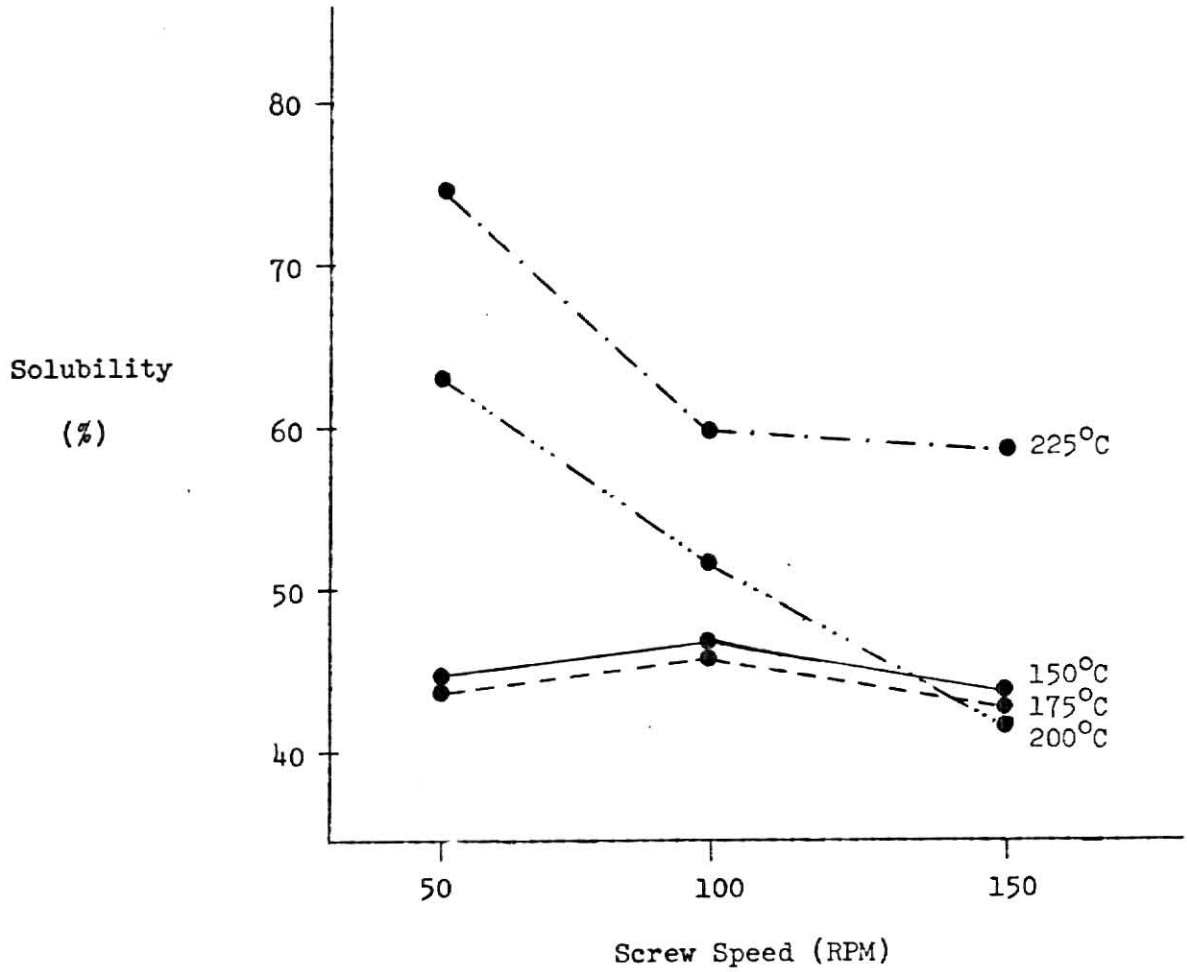


Figure 15

Blue Agtron Reading VS Moisture

(Averaged Over All Screw Speeds)

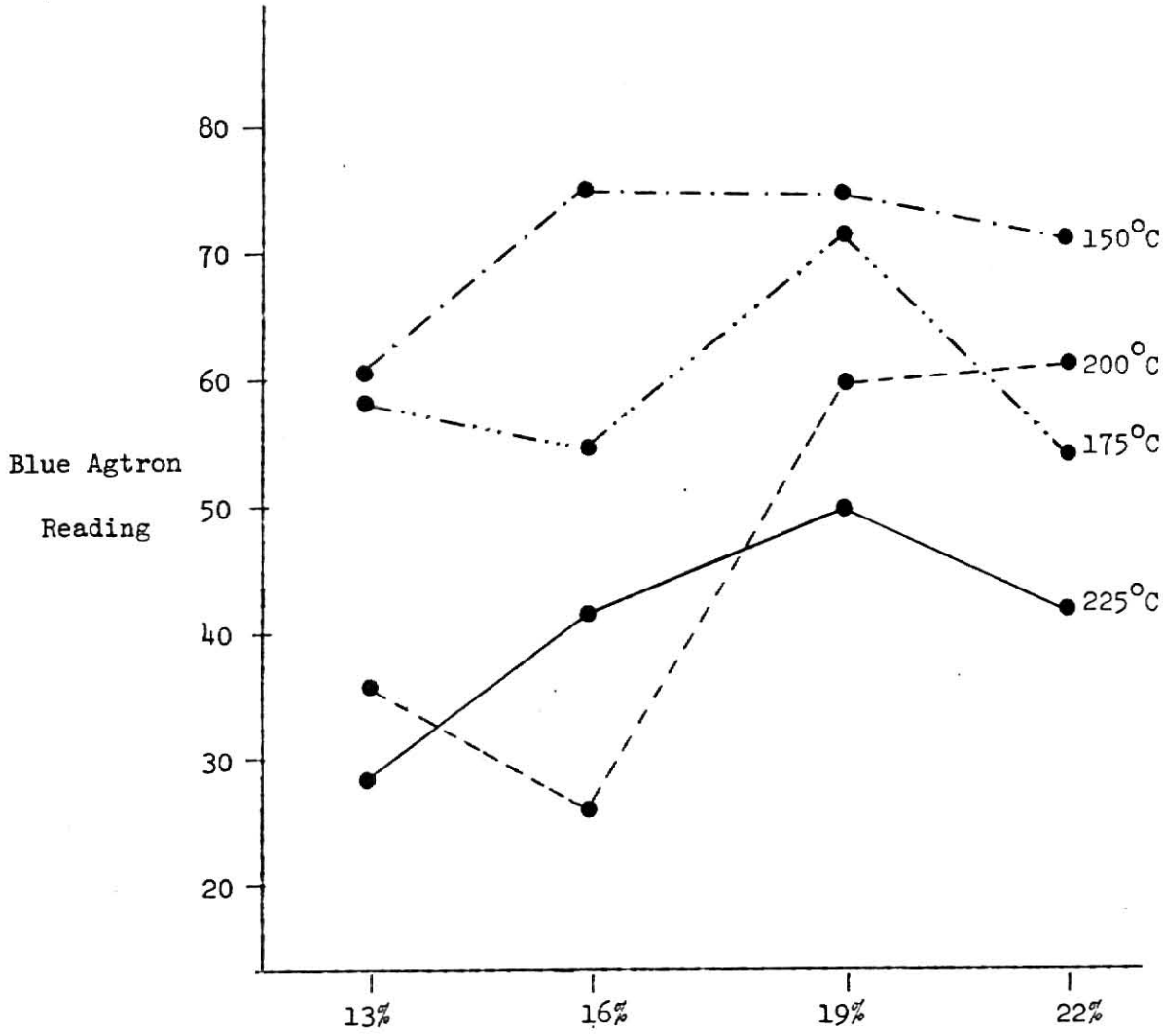


Figure 16

Green Agtron Reading VS Moisture

(Averaged Over All Temperatures & Screw Speeds)

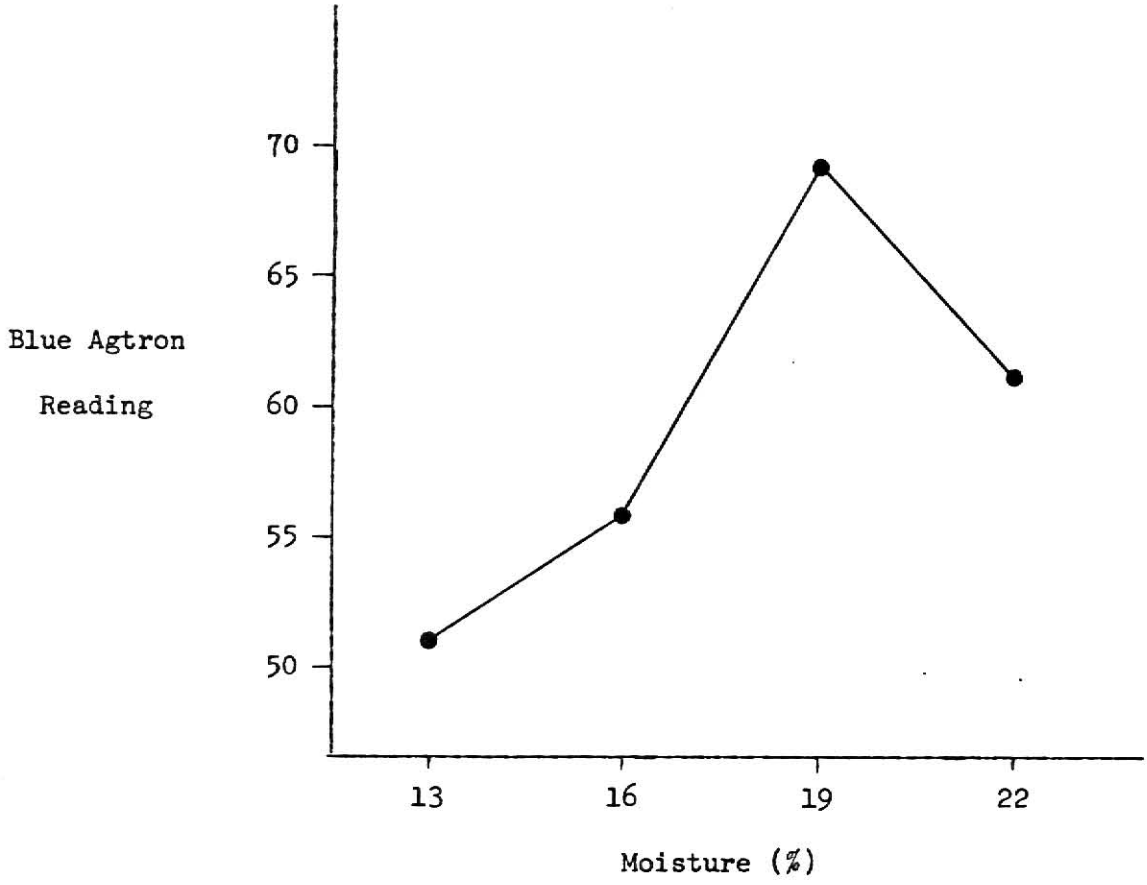


Figure 17

Blue Agtron Readings VS Temperature

(Averaged Over All Screw Speeds)

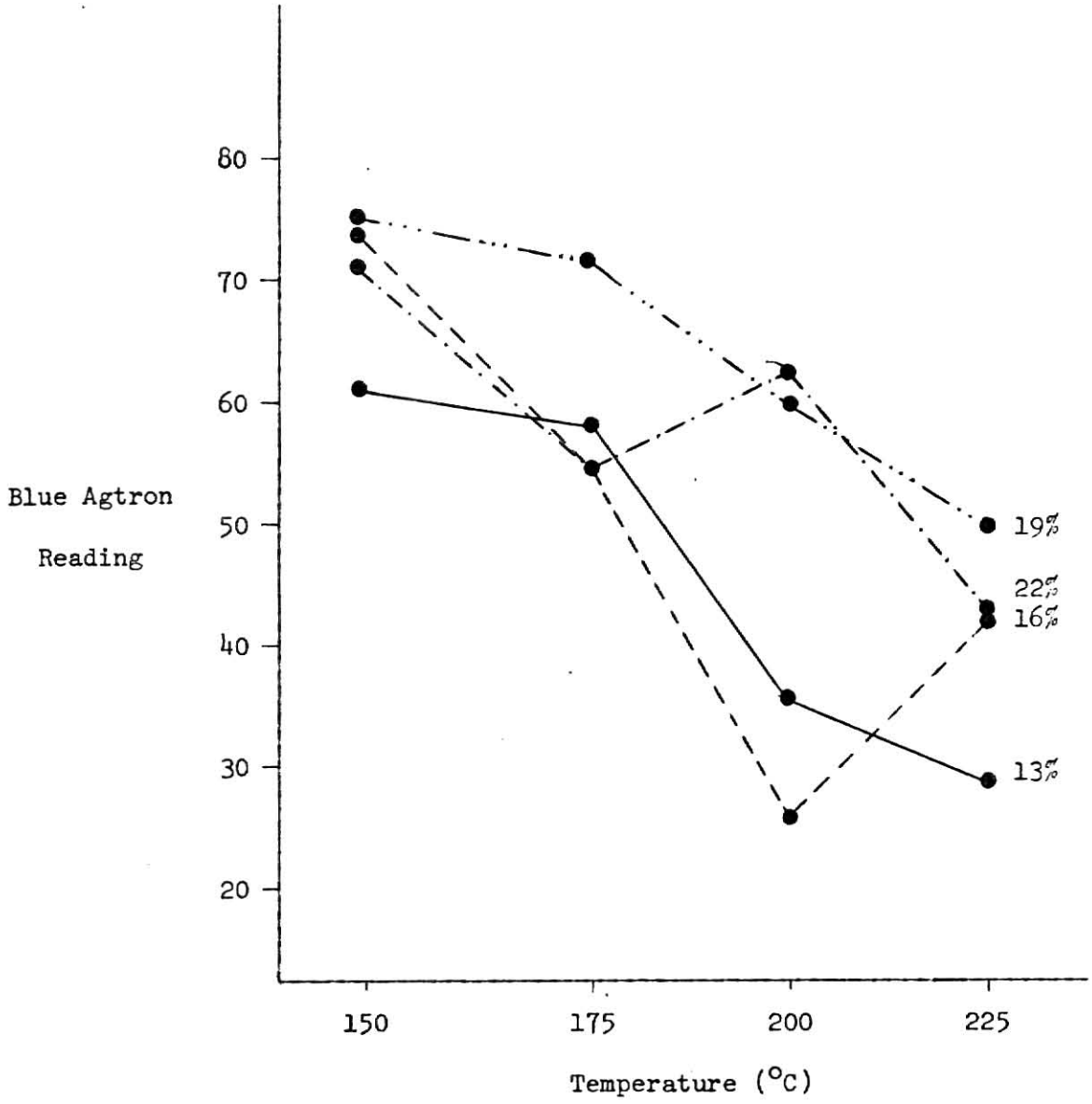


Figure 18

Green Agtron Readings VS Temperature

(Averaged Over All Moisture Levels & Screw Speeds)

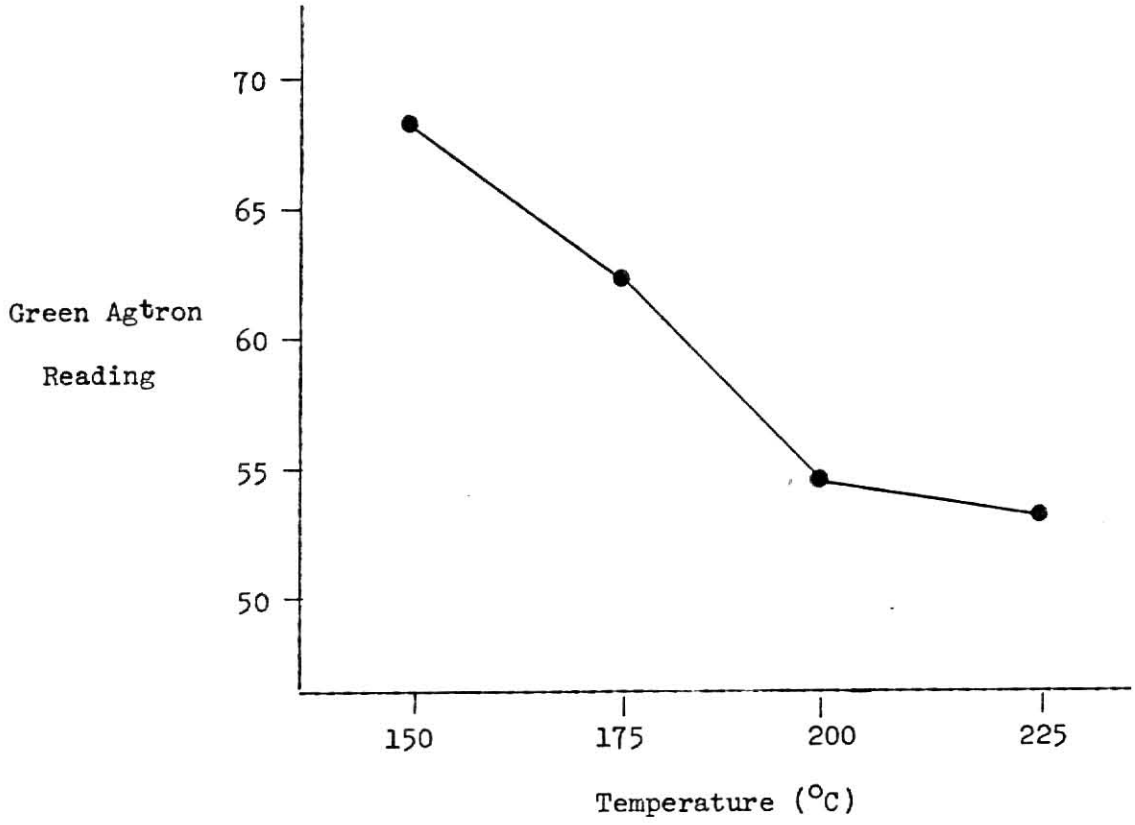


Figure 19

Blue Agtron Readings VS Shear Rate

(Averaged Over All Moisture & Temperature Levels)

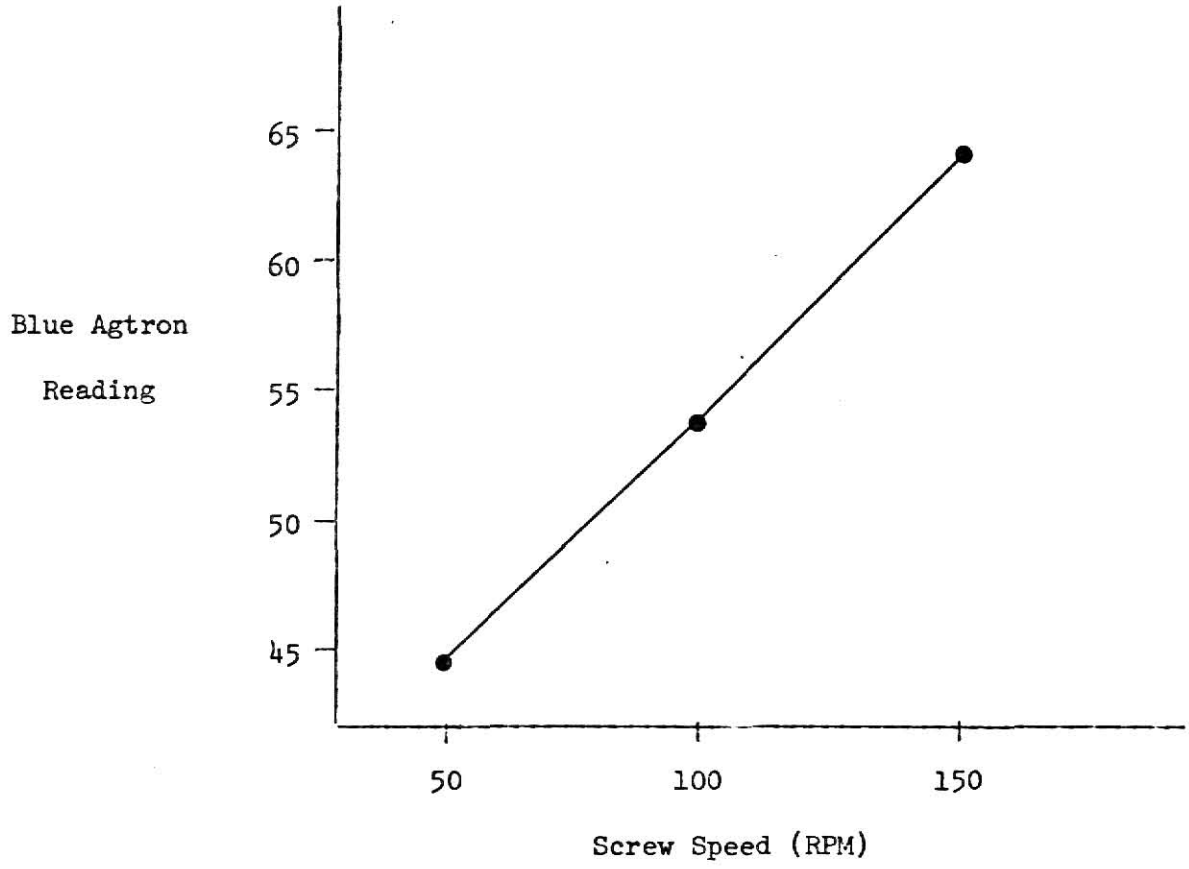


Figure 20

Green Agtron Readings VS Shear Rate

(Averaged Over All Moisture & Temperature Levels)

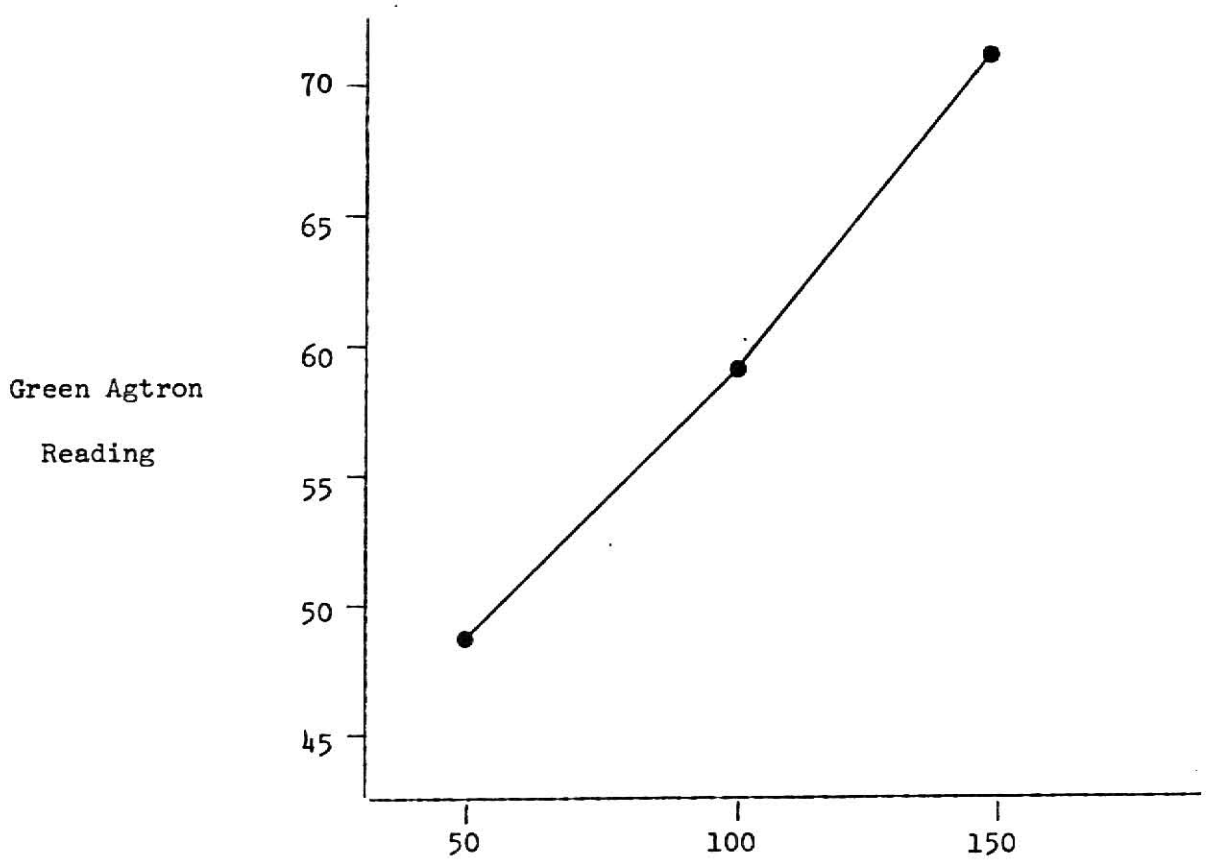


Figure 21

SEM of Raw Starch
Magnification - 920x

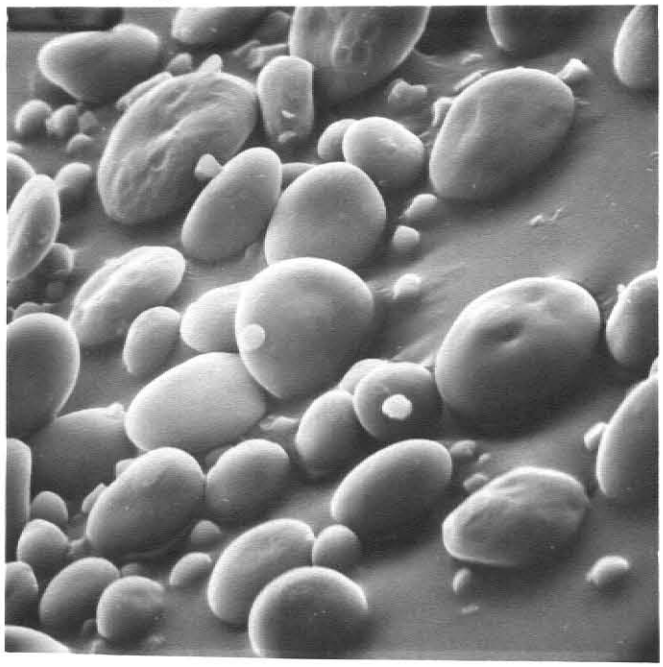


Figure 22

SEM of Extruded Starch Simple X-042
Magnification - 13x

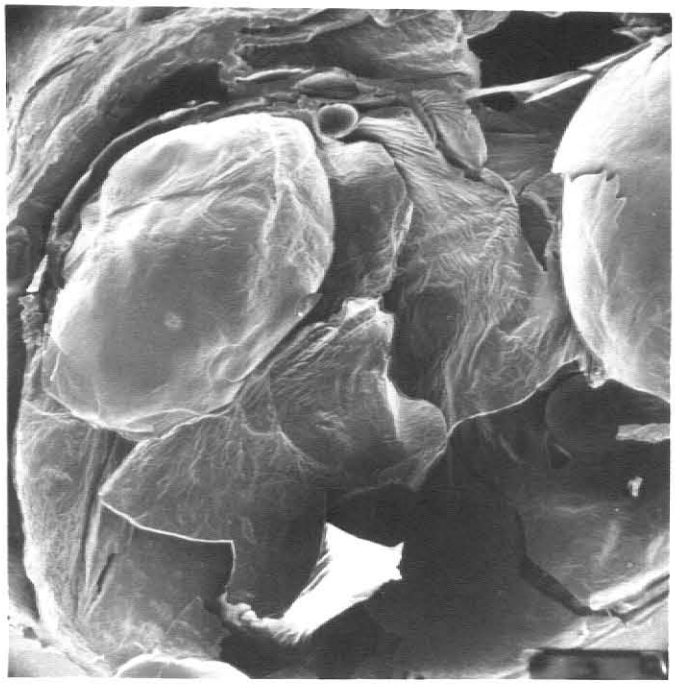


Figure 23

SEM of Extruded Starch Sample X-042
Magnification - 20x

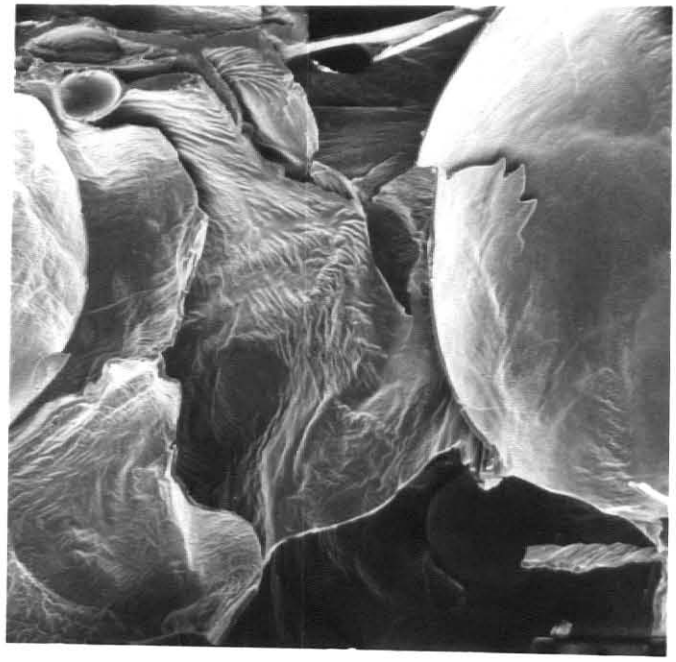


Figure 24

SEM of Extruded Starch Sample X-042
Magnification - 64x

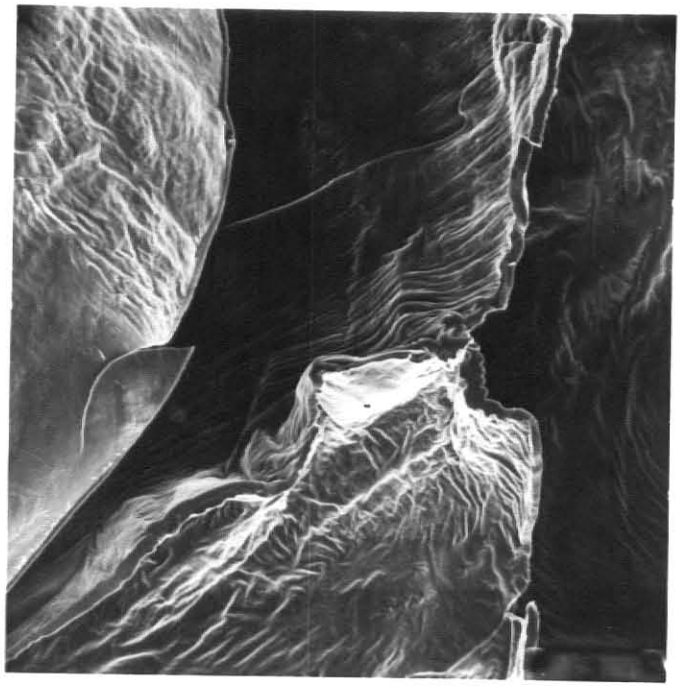


Figure 25

SEM of Extruded Starch Sample X-042
Magnification - 208x

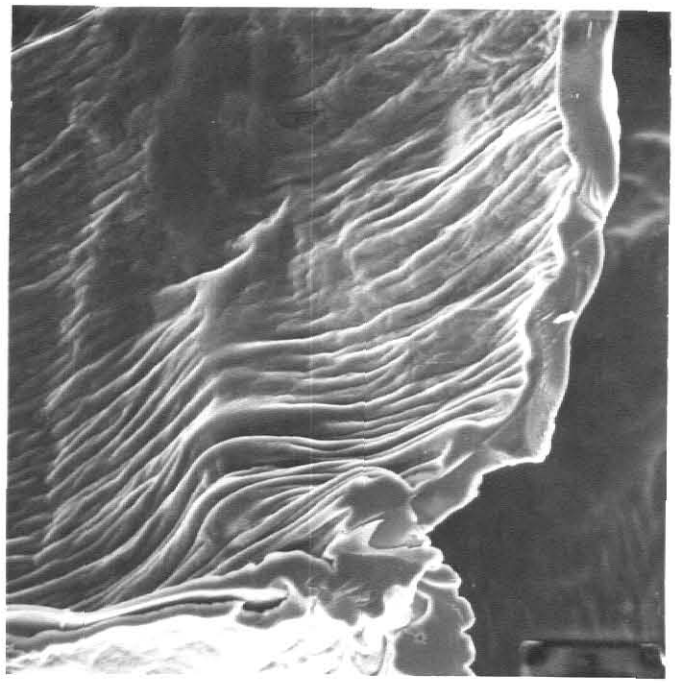


Figure 26

SEM of Extruded Starch Sample X-042

Magnification - 208x

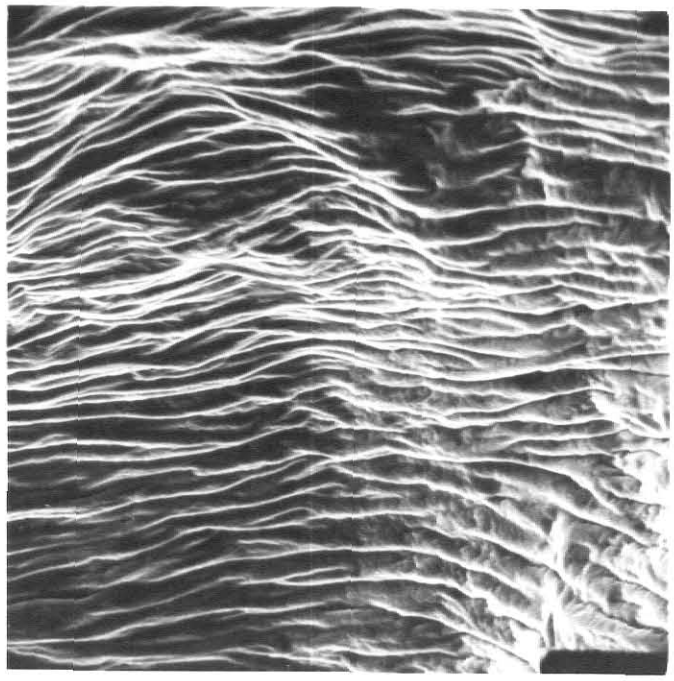


Figure 27

SEM of Extruded Starch Sample X-042
Magnification - 206x

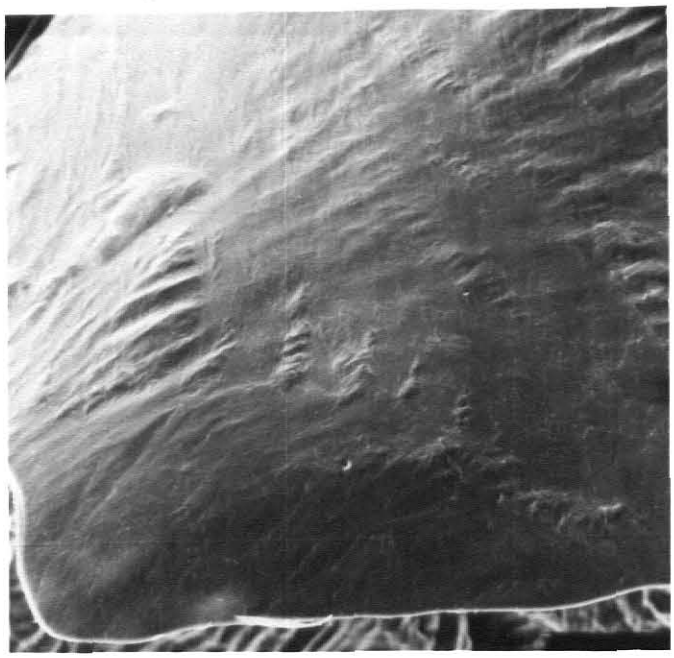


Figure 28

SEM of Extruded Starch Sample X-073
Magnificatin - 12.5x

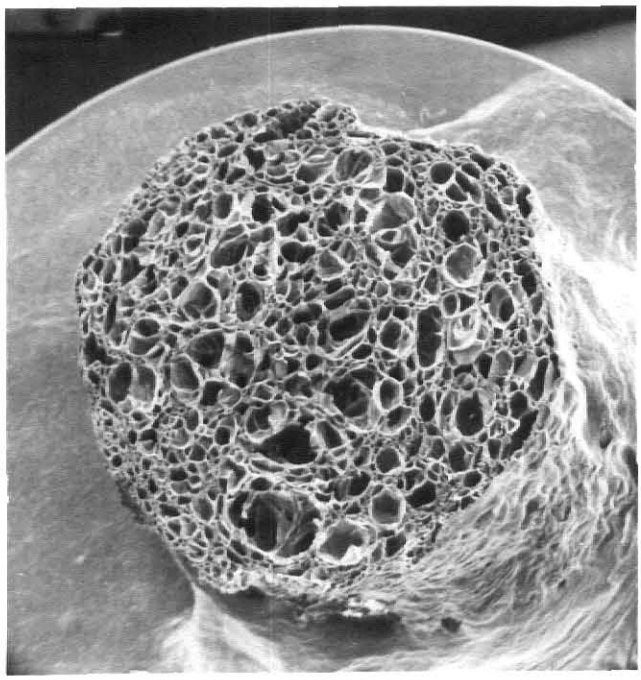


Figure 29

SEM of Extruded Starch Sample X-073
Magnification - 64x

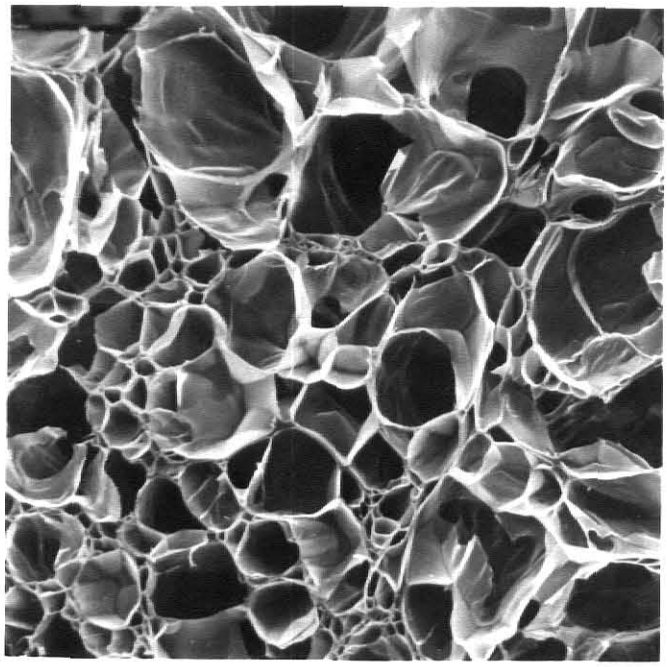


Figure 30

SEM of Extruded Starch Sample X-073
Magnification - 208x

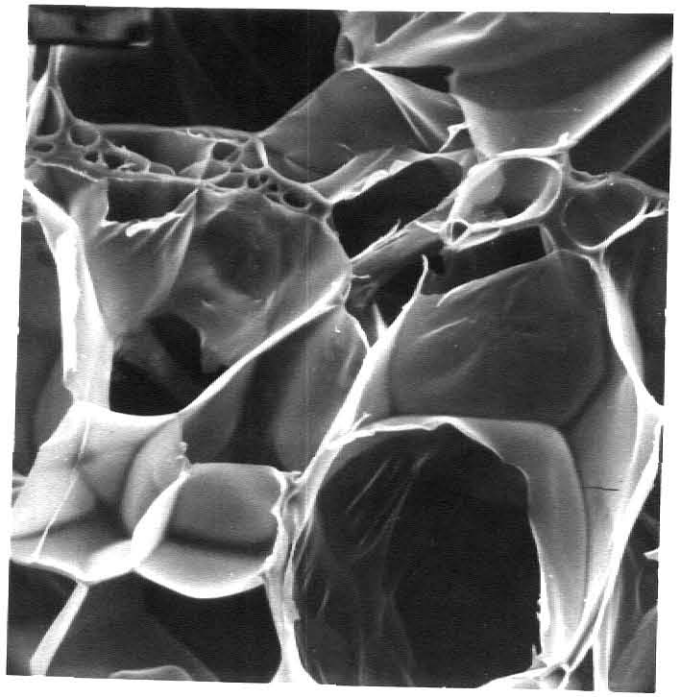


Figure 31

SEM of Extruded Starch Sample X-073
Magnification - 3000x

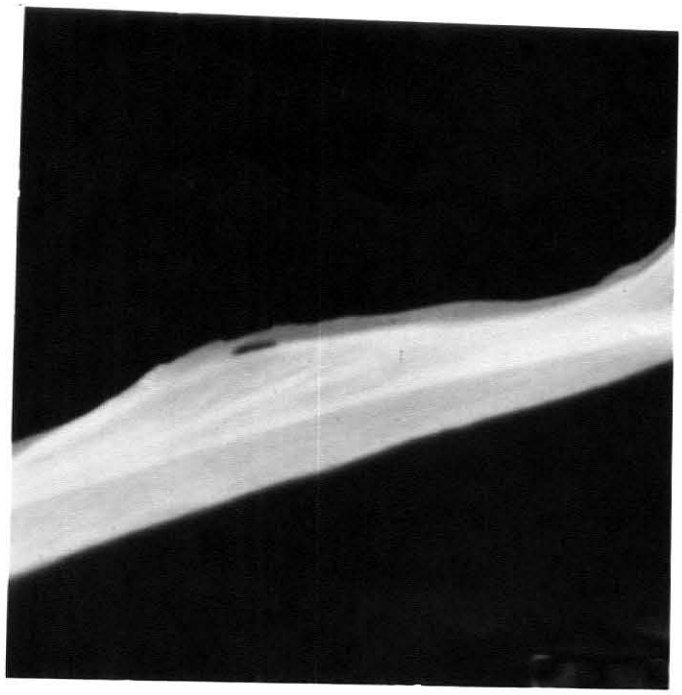
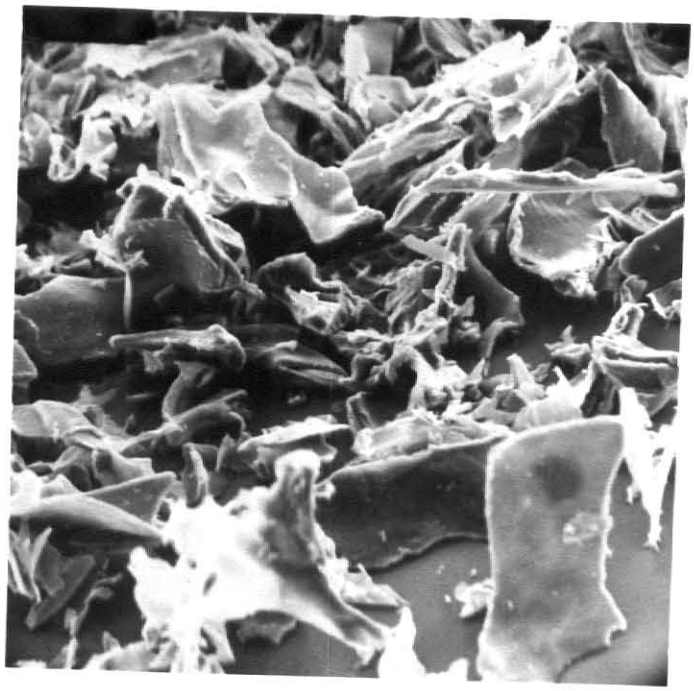


Figure 32

SEM of Extruded Starch Sample X-073
Magnification - 184x



APPENDIX I

Viscosity (centipoise)

Temperature	Screw Speed	Moisture 13%	16%	19%	22%
	150 RPM	12.2	11.6	12.2	9.5
225°C	100	13.2	11.2	11.6	9.0
	50	N.A.	9.2	8.7	7.6
	150	20.2	20.2	19.2	14.2
200°C	100	19.6	22.2	17.0	14.7
	50	N.A.	17.4	19.2	10.7
	150	23.6	23.4	21.2	22.9
175°C	100	23.0	23.2	18.2	22.2
	50	N.A.	26.7	20.4	27.2
	150	21.6	24.6	18.0	29.2
150°C	100	21.2	22.4	18.1	24.4
	50	N.A.	20.3	15.4	23.3

Raw Wheat Starch		86.6
Two Commercial Pergelatinized	1	60.3
Wheat Starches	2	55.2

N.A. - Not Available

Expansion (inches)

Temperature	Screw Speed	Moisture 13%	16%	19%	22%
	150 RPM	.3125	.2594	.2156	.1969
225°C	100	.3219	.2219	.1844	.1718
	50	N.A.	.1718	.2157	.1968
	150	.4062	.3844	.3906	.2031
200°C	100	.4250	.3656	.3563	.2000
	50	N.A.	.3500	.3125	.2000
	150	.4562	.4500	.4125	.3250
175°C	100	.4250	.4031	.3906	.3563
	50	N.A.	.3688	.3501	.3156
	150	.4031	.4156	.3968	.2844
150°C	100	.4156	.3563	.3969	.3281
	50	N.A.	.4250	.3562	.3344

N.A. - Not available

pH

Temperature	Screw Speed	Moisture 13%	16%	19%	22%
	150 RPM	6.40	5.52	5.82	5.56
225°C	100	6.30	5.32	5.66	5.48
	50	N.A.	5.15	5.30	5.20
	150	6.81	6.66	6.34	6.46
200°C	100	6.55	6.68	6.18	6.20
	50	N.A.	6.61	6.30	5.70
	150	6.90	7.20	6.85	6.97
175°C	100	6.84	7.05	6.66	6.96
	50	N.A.	7.03	6.64	6.92
	150	6.86	7.26	6.95	7.35
150°C	100	6.80	7.01	7.05	7.16
	50	N.A.	7.25	6.94	7.28

Raw Wheat Starch		6.76
Two Commercial Pregelatinized	1	5.29
Wheat Starches	2	3.72

N.A. - Not Available

Water Solubility (percent)

Temperature	Screw Speed	Moisture 13%	16%	19%	22%
	150 RPM	83.64	46.77	45.38	61.86
225°C	100	81.71	55.52	42.52	60.98
	50	N.A.	67.71	59.68	74.01
	150	56.70	51.58	31.27	28.45
200°C	100	73.47	57.04	39.43	39.34
	50	N.A.	66.16	52.16	52.27
	150	48.95	49.45	44.91	30.14
175°C	100	47.74	48.19	52.13	35.36
	50	N.A.	45.71	49.90	33.59
	150	45.23	42.80	51.37	36.11
150°C	100	55.53	40.97	49.19	40.67
	50	N.A.	46.09	46.83	36.56

Raw Wheat Starch		0.01%
Two Commercial Pregalatinized	1	9.50%
Wheat Starches	2	13.10%

N.A. - Not Available

Blue Agtron Color

Temperature	Screw Speed	Moisture 13%	16%	19%	22%
	150 RPM	40.0	44.8	56.9	54.0
225°C	100	28.5	37.3	49.8	56.5
	50	N.A.	45.0	43.0	18.3
	150	56.0	42.9	69.0	72.0
200°C	100	31.0	29.8	67.0	64.0
	50	N.A.	9.1	44.0	48.8
	150	72.0	69.0	81.0	66.0
175°C	100	55.2	48.8	77.7	53.0
	50	N.A.	46.5	56.5	46.0
	150	78.0	82.0	77.5	72.0
150°C	100	46.5	74.3	86.0	60.0
	50	N.A.	67.5	62.5	80.0

Raw Wheat Starch		100.0 +
Two Commercial Pregelatinized	1	90.0
Wheat Starches	2	82.3

N.A. - Not Available

Green Agtron Color

Temperature	Screw Speed	Moisture 13%	16%	19%	22%
	150 RPM	51.5	52.4	67.0	68.0
225°C	100	33.4	51.9	66.0	72.0
	50	N.A.	59.0	55.0	35.5
	150	65.0	57.0	82.0	79.0
200°C	100	35.8	40.0	78.0	70.0
	50	N.A.	12.8	57.0	55.0
	150	82.0	75.0	85.0	74.0
175°C	100	52.0	54.9	88.5	56.0
	50	N.A.	53.3	44.0	44.0
	150	81.0	80.0	68.2	69.5
150°C	100	51.5	65.0	86.5	49.0
	50	N.A.	69.0	56.5	78.0

Raw Wheat Starch		100.0 +
Two Commercial Pregelatinized	1	100.0 +
Wheat Starches	2	100.0 +

N.A. - Not Available

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EXTRUSION COOKING OF WHEAT STARCH

by

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B.S., Kansas State University, 1971

AN ABSTRACT OF A THESIS

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Manhattan, Kansas

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ABSTRACT

An experimental high temperature-short time extrusion cooker was used in the extrusion of wheat starch at four moisture levels, four temperature levels, and three screw speeds. The following five properties of the extruded wheat starch were examined: solution viscosity, pH, expansion, water solubility, and color formation. A Least Squares Statistical Analysis of Variance with unequal subclasses was used to determine the statistical significance of the three independent variables and two of their two-way interactions.

As temperature increased solution viscosity and pH decreased while water solubility and color formation increased. Decreasing moisture increased expansion but a strong interaction with temperature was observed. The effects of decreasing screw speed showed decreased solution viscosity, increased water solubility, and a linear increase in color formation in the starch extrudate. All samples were considered completely gelatinized and there was no evidence in the formation of undigestible bonds not susceptible to the enzyme glucoamylase.