VISCOMETRIC PROPERTIES OF CORN
WATER SLURRIES DURING
COOKING FOR ETHANOL
PRODUCTION

By

ALI KHAKBAZ

Bachelor of Science
in Agricultural Engineering
Oklahoma State University
Stillwater, Oklahoma
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Thesis Approved:

Bobby L. Clary

Thesis Adviser

A. D. Barefoot

James E. Garton

Norman N. Durham

Dean of the Graduate College

PREFACE

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CHAPTER I

INTRODUCTION

Interest in alcohol fuels as an alternate energy source and as a potential new agricultural industry has expanded enormously in the last few years.

Fuel ethanol may be produced from sugar and starch base feedstocks. In order that the yield of ethanol be maximized, it is imperative that process reaction conditions such as pH and temperature be optimized and maintained within narrow limits (25).

Ethanol production consists of three major stages: the formation of a solution of fermentable sugars, saccharification, the fermentation of sugars to ethanol, and the distillation of ethanol (8). Different raw materials require variations in the initial part of the first production step -- the processing of the raw material to ultimately obtain fermentable sugars. Grain or other starch containing materials first require a reaction of starch with water (hydrolysis) in the presence of enzymes to produce a simple sugar solution. The second step -- fermentation -- is the same for all feedstocks and utilizes yeast to convert sugar to ethanol and carbon dioxide. The fuel alcohol is then obtained by distillation of the ethanol-water mixture (24).

Energy balances during ethanol production is still widely disputed. Recent advances in process technology, along with well designed plants enable one to produce anhydrous ethanol with a process energy input of approximately 40,000 Btu per gallon of ethanol (7). An additional 40,000 Btu per gallon (7) are required to grow, harvest and transport the grain feedstock, resulting in total production energy requirements of 80,000 Btu per gallon (7). The energy contained in a gallon of anhydrous ethanol is 84,000 Btu per gallon (7).

It is, therefore, necessary to find ways of reducing energy input in production of ethanol. Parkinson (25) and Hartline (12) looked into some alternatives to conventional distillation processes; as an example a solvent extraction process for removing water from ethanol promises to use as little as 13% as much energy as conventional distillation.

Downs and Clary (6) suggested that half of the total energy requirement for ethanol production can come from non-liquid fuel sources such as wood and wood waste products, coal and crop residues. Waste heat streams which exit at several places can be passed through heat exchangers to provide pre-heating for other parts of the process.

When an aqueous suspension of granular starches is heated above the gelatinization temperature[1], the granules

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[1] Starch gelatinization is usually described as the rapid change from dispersion to paste that is observed when an aqueous starch slurry is heated (37).

become highly hydrated and swell to many times their original volumes. Continued heating, particularly in the presence of shear, produces cooked pastes which are mixtures of swollen granules, granule fragments and molecularly dispersed starch molecules leached from the granules (16).

Granule swelling and disintegration during cooking are accompanied by significant changes in the viscosity and other rheological properties of the slurry (36).

Objective

The objectives of this study are:

1. To evaluate the viscometric properties of ground corn mash during cooking for ethanol production.
2. To evaluate the shear stress-strain rate relationship at peak point, 165°F (73.9°C), and at 200°F (93.3°C).

CHAPTER II

LITERATURE REVIEW

An important factor in determining non-Newtonian slurry viscosity is the solid volume fraction. The slurries, tend to be Newtonian for low solid concentrations (13,14). The volume fraction of solid has the strongest influence on the apparent viscosity of slurries (13, 32, 38, 39). Other factors which may also effect slurry viscosities, are the shape and size distribution of suspended particles, temperature, surface properties, electrical charges and nature of the flow fields (33).

Harrison (10) was the pioneer in determining viscosity of starch granules. He concluded that the stiffening powers of different starches correspond qualitatively to the amount of water they absorb (10). Anker and Geedes (2) observed and explained the relationship between maximum viscosity and initial starch concentration by assuming: (a) that during the gelatinization process, the ungelatinized granules A form highly swollen gelatinized granules B which are then eventually ruptured as a result of shearing action, thereby losing most of their incorporated water and forming relatively nonhydrated disintegrated granules C; (b) that the viscosity increment due to A and to C is small or insignificant as compared with the viscosity increment due to B; (c) that the viscosity concentration relationship with respect to B is approximately described by the Arrhenius equation: i.e. $\log \mu_r = K[B]$ (where [B] denotes the concentration of B); (d) $\mu_s >> \mu_o$, where μ_s = viscosity of solution and μ_o = viscosity of solvent. The relative viscosity μ_r will then be approximately proportional to μ_s and the value of μ_s can be substituted for μ_r in the Arrhenius equation; that is $\log \mu_s = K[B]$; (e) that the process $A \rightarrow B$, involving the taking up of water by the granule, is a first order process. At the end of a given time, the amount of B present will be directly proportional to the initial amount of A present. This process can be expected to be of the first order as long as there is sufficient water present so that no competition occurs between granules for water; (f) that the process $B \rightarrow C$, involving the rupture or disintegration of the gelatinized granules is a second - or higher - order process. This is expected since the rate of disintegration is a function of the shearing action on the swollen granules, and the intensity of this shearing action is a function of the viscosity, which in turn is a function of the concentration of B.

On the basis of these assumptions, the amount of B formed in a given time after initiation of the process is proportional to the initial concentration of A; however, as the initial concentration of A is increased, the maximum amount of B is reached in a shorter time, and the value of the maximum attained for B varies logarithmically with the initial concentration of A (or to the amount of B formed in each comparable unit of time after the process A ⟶ B is initiated):

thus, $[B]_{max} = K \log [A]_{initial}$ (1)

and $\log \mu_{smax} = K'[B]_{max}$ (2)

then $\log \mu_{smax} = K'K \log [A]_{initial}$; (3)

meaning that the maximum viscosity of solution versus initial starch used is a straight line on log-log coordinates.

Starch

Most starches are mixtures of two polymers, amylose and amylopectin. Amylose is a linear $(1 \rightarrow 4)$-α-D glucan usually having a degree of polymerization (d.P) of 400, while amylopectin is a branched D-glucan having mostly α-D-$(1 \rightarrow 4)$ linkages, but with 4% of linkages of the α-D-$(1 \rightarrow 6)$ type as shown in Figure 1. This branched polymer, designated amylopectin, consists of short, amylose like chains of d.P. 12 to 50, linked into a branched structure (18).

Starches of the various plants differ with respect to their proportions of amylose and amylopectin. For example, the starch in corn (maize) of the variety most commonly grown in the United States contains 26% amylose and 74%

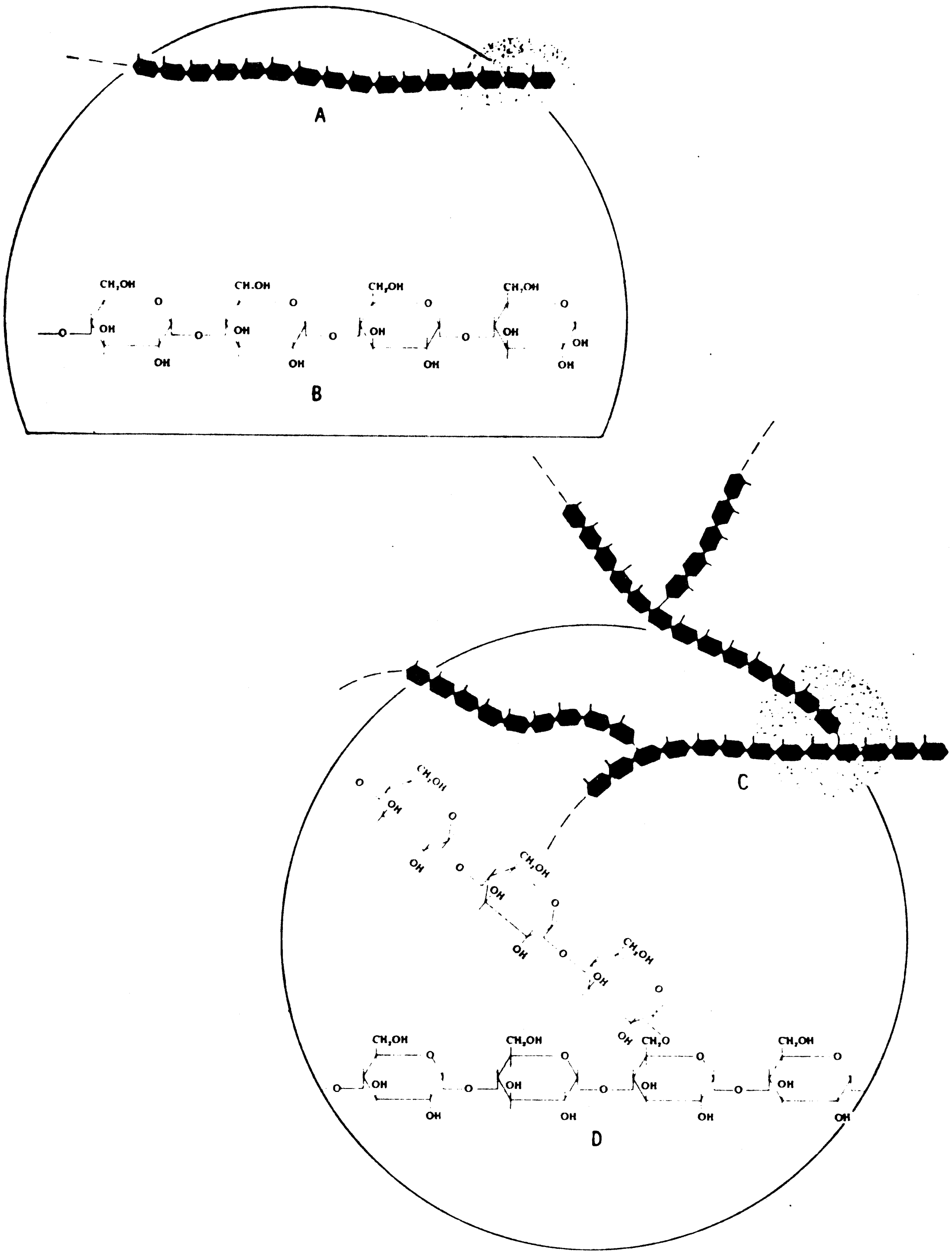

Figure 1. Structure of the Amylose and Amylopectin Component of Starch. A: Diagram of a Portion of an Amylose Molecule; B: Enlarged View of the Shaded Section Showing Chemical Formula; C: Diagram of a Portion of an Amylopectin Molecule; D: Enlarged View of Shaded Area Showing Chemical Formula. (27)

amylopectin (18).

Starch usually occurs in plants as discrete granules. In corn these are irregular polyhedra having a width of 10 to 20 μm (18). The starch granule poses many challenging problems. Little is known of the way in which the starch material - amylose, amylopectin and the intermediate material - is combined together to form the resultant granule. The problem faced in understanding the structure of granules are best epitomized in Figure 2-4 (9), which show scanning electron micrographs of starch from maize and its genotypes. Granule shape and size depend on the apparent amylose content. The typical angular granules of normal maize (Figure 2) decrease as amylose content increases from 27% to 50% with more rounded forms become common (Figure 3). When apparent amylose content of 70% is attained (Figure 4), sausage shaped granules appear (9, 19).

It has been recognized in recent years that under certain conditions an atom of Hydrogen is attracted by rather strong forces to two atoms, instead of only one, so that it may be considered to be acting as a Hydrogen bond between them. It is now recognized that -- the hydrogen bond is largely ionic in character and is formed only between the most electromagnetic atoms (26).

Experimental difficulties have prevented the direct application of physical methods to find H-bonds in polysaccharides such as starch and cellulose but indirect evidence of such association is so convincing that hydrogen bonding

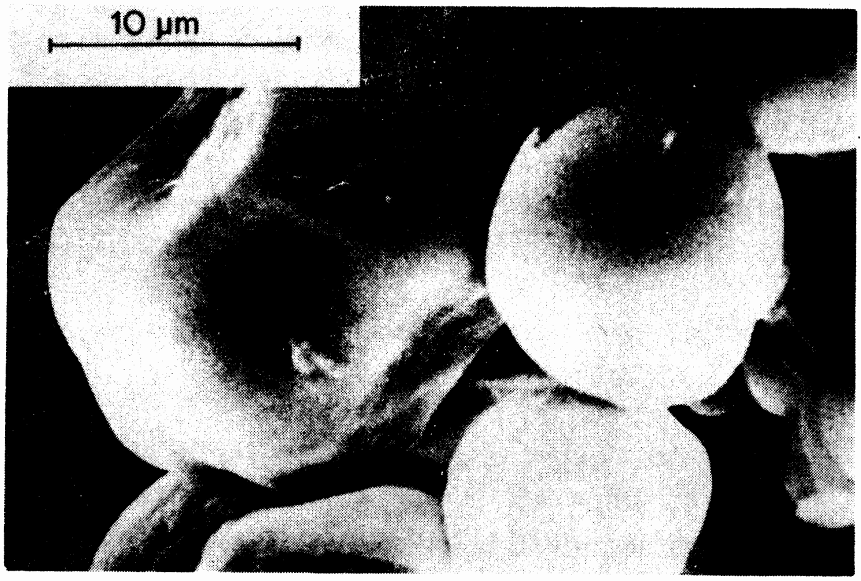

Figure 2. Scanning Electron Micrograph of Granules of Normal Maize Starch Showing Typical Angular and Rounded Granules (9)

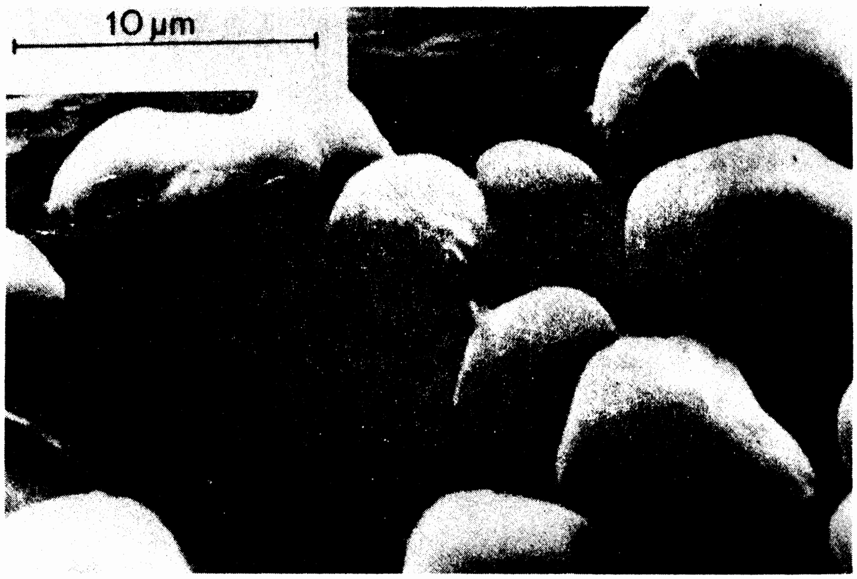

Figure 3. Scanning Electron Micrograph of Granules of Maize Starch with a Reputed Amylose Content of 50% (Amylon 50) Showing Unusual Rounded Granular Forms (9)

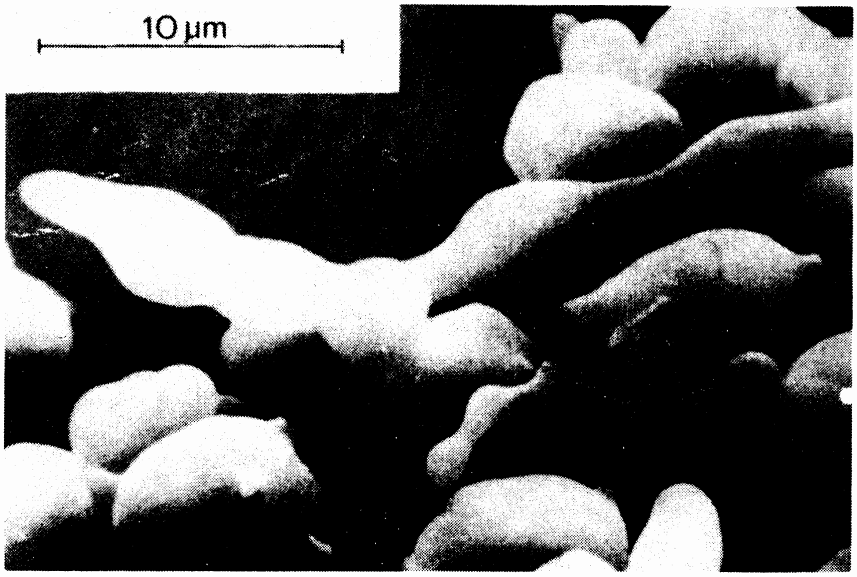

Figure 4. Scanning Electron Micrograph of Granules of Maize Starch with a Reputed Amylose Content of 70% (Amylon 70) Showing Bizarre Granular Forms (9)

is now generally accepted (4). Taylor (37) was probably among the first to recognize its important role in the field of starch chemistry. It was his opinion that the study of starch chemistry is incomprehensible without the postulation of micellar association through hydrogen bonding.

Caeser (4) outlined the behavior of starch in an aqueous dispersion, and postulated the association of primary valance chains through H-bonds. In Figure 5 the association of two molecules of starch in the absence of water (I) and the association through water (II) is indicated schematically.

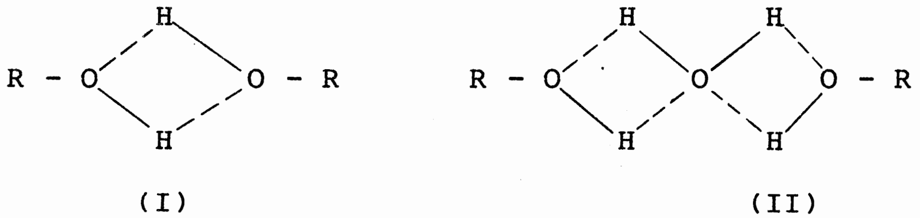

Figure 5. Association of Hydrogen Bonds in Starch; (I) Inactive Form; (II) Active Form. The Hydrogen Bonds are Represented by Dots.

In a polyhyroxylated compound such as starch, the arrangement would be expected to reduce the activity of hydroxyl groups. When starch is desiccated or thoroughly retrograded the internal arrangement approaches an association of type (I) in which neighboring OH groups directly associate or are bonded with one another. In type (II) which may be designated as starch in a normal state, neighboring OH groups are linked through one or more molecules of H_2O which pry them apart and render them more reactive (the O - H --- O distance is 2.76A (26)). Retrograding the starch granules resulted in moisture loss in excess of 10 percent. The assumption of association through hydrogen bonding infers that easily expelled water is loosely bonded to the exterior of micelles and the balance is bonded more or less intramicellarly as in (II).

Gelatinization of Starch

A normal starch granule when placed in cold water swells to a limited extent, perhaps 10% in diameter or 30 to 35% in volume (1, 12, 29). On the other hand, in hot water, the granule gelatinizes, taking up to 10 or more times its weight of water with the hydroxyls of a gelatinized starch molecule holding large quantities of water by hydrogen bonding (4).

In the absence of mechanical action, relatively little granule disintegration and solubilization of the starch takes place (2). From studies of viscosity changes which occur in corn and potato starch pastes on heating and stirring, Katz (15) concluded that the heat gelatinization curve is a result of two opposing factors: One, the progressive swelling and hydration of the starch granule (which increases viscosity), and the other the breakdown of the vesicle walls (which decrease viscosity). Schoch (30) pointed out that the viscosity of a boiled starch paste must be due largely to the presence of swollen aggregates or fragments of granule structure since the viscosity markedly decreases upon autoclaving or violent mechanical agitation.

As starch concentration increases, there is an appreciable decrease in the temperature at which the viscosity shows a measureable change, a marked increase in maximum viscosity, and a slight decrease in the temperature of the paste at which the peak viscosity is registered. Morever, the peak viscosity is more abrupt, and the subsequent decrease in viscosity is more rapid (2). Figure 6 shows the Brabender curves at a variety of starch concentrations as replotted on rectangular coordinates (17). There are five successive points of significance on these curves:

A - Peak viscosity, irrespective of the temperature at which the pasting peak is attained.

B - The viscosity when the paste reaches a temperature of 95°C.

C - Viscosity after cooking for one hour in the Brabender at 95°C.

D - The viscosity when the cooked paste is cooled to 50°C.

E - The final viscosity after stirring in Brabender for one hour at 50°C.

Figures 7 and 8 shows the viscosities at five different

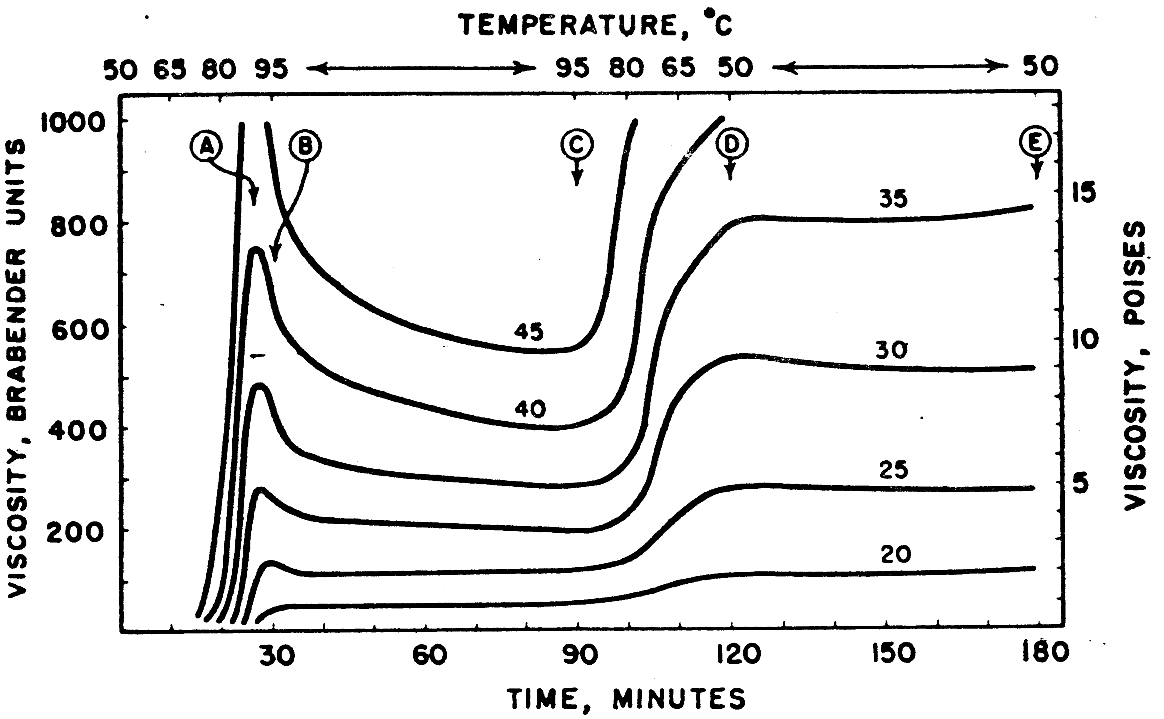

Figure 6. Brabender Curves of Thick-boiling Corn Starch. Letter Keys Indicate Viscosity Reference Points. Numerals Indicate Starch Concentration, in g. per 500 ml (17)

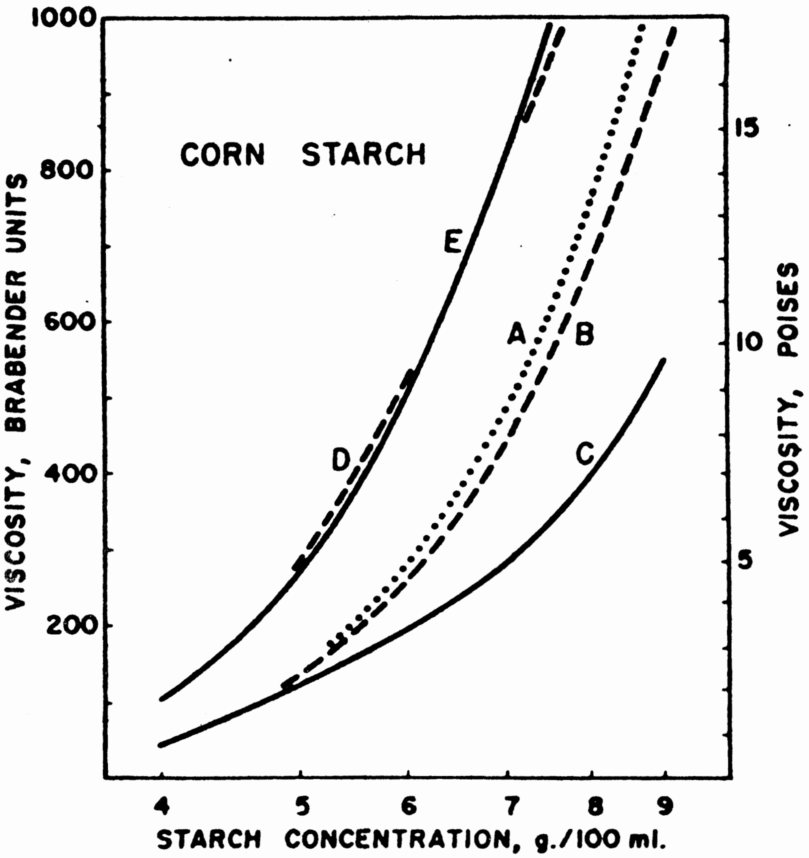

Figure 7. Viscosities Values of Heating Unmodified Corn Starch at Five Different Critical Points (17)

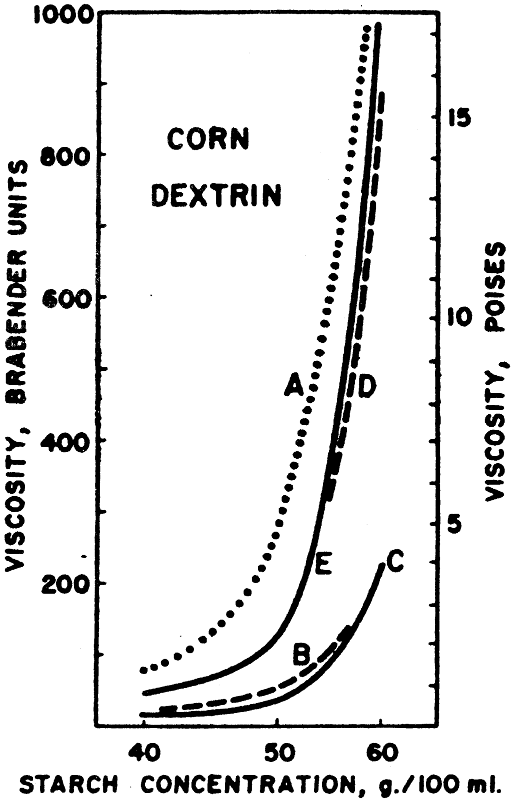

Figure 8. Viscosity Values of Heating Commercial Corn White Dextrin at Five Different Critical Points (17)

critical points for commercial unmodified corn starch and commercial corn white dextrin (17). Figure 9 (9) shows the typical pasting behaviour of starches from the genotypes of maize: normal maize, waxy maize (with no amylose content), and amylomaize (with a reputed amylose content of 50%). As temperature of water is increased, granules swell to impinge on each other and increase viscosity of the starch paste. This process continues until peak "viscosity" is reached, for at this point cohesive forces in the original granule structure become excessively weakened and structure of the paste - and in consequence, the observed viscosity - collapses as integrity of the granule is lost. Figure 8 shows that waxy maize starch swell rapidly to give a high peak viscosity, but the intermolecular forces in this granule are weak and break down rapidly on further cooking. On cooling, however, there is very little "set back" because of absence of amylose. Normal maize starch takes a longer time to reach a much lower peak viscosity; the extent of breakdown of structure on prolonged stirring at high temperature is similar, but the degree of set back is large because of aggregation of amylose. In contrast, the amylomaize starch registers no swelling at all (9).

Viscosity

The measurement of viscosity is of considerable importance in both industrial production and fundamental science. Viscosity is the quantity that determines the forces to be

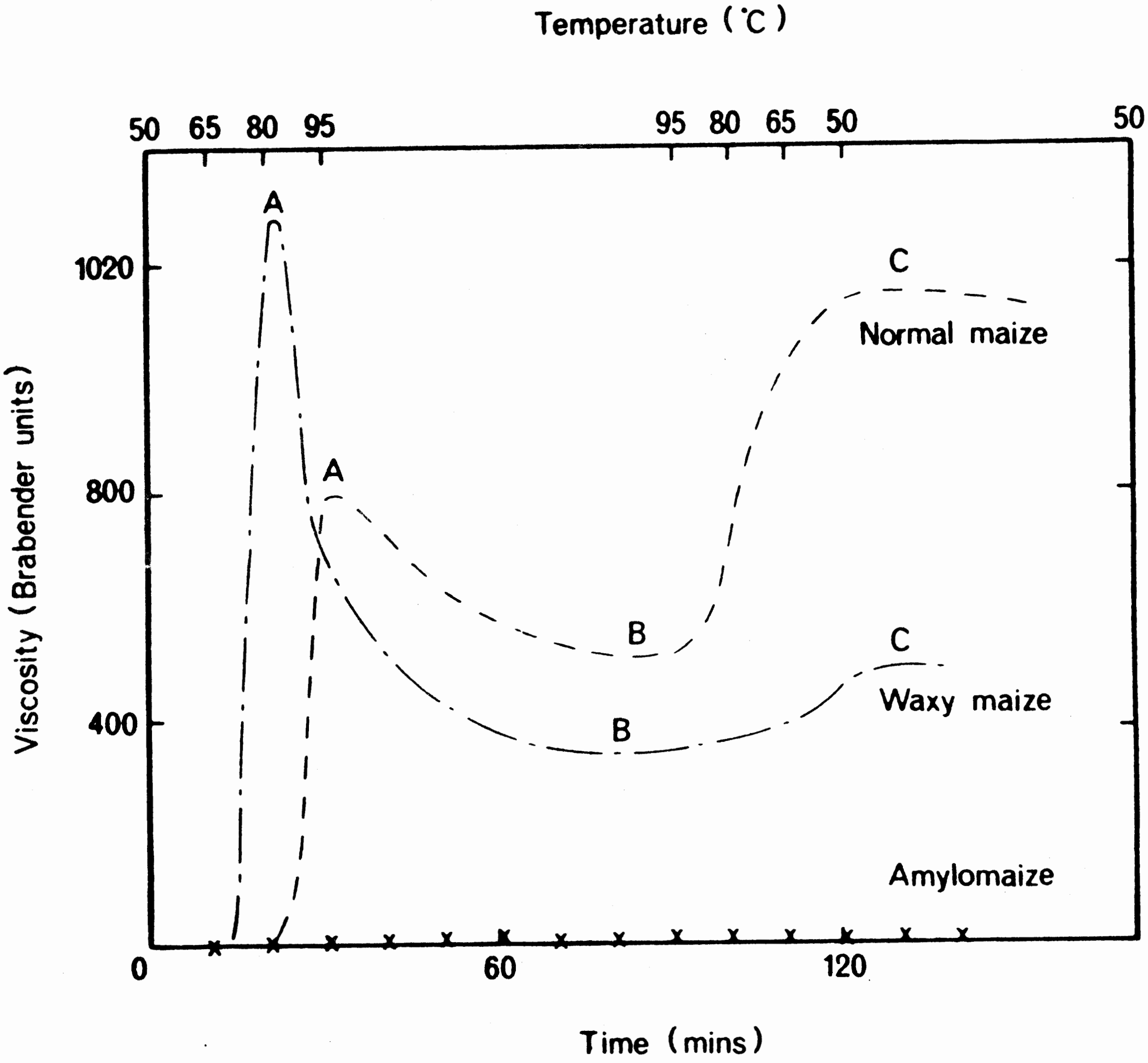

Figure 9. Typical Behavior of Paste Viscosity, as Function of Temperature and Time when Measured in the Brabender Amylograph, for Normal Maize, Waxy Maize and Amylomaize (Amylon 50). A - the "Peak Viscosity", the Maximum Viscosity Achieved on Heating the Dispersion; B - the Extent of Breakdown of Paste Structure After Prolonged Stirring at Elevated Temperature; C - the Amount of "Set Back", i.e. the Development of Aggregated Structures on Cooling (9)

overcome when fluids are used in pipelines, bearings, etc. and it controls the flow of liquid in such processes as spraying, injection mouldings, extrusion and surface coating. Viscosity has an important bearing on the mixing and heat transfer characteristics of fluids. Viscosity measurement has also proved a valuable tool for the physical chemist since the viscosity coefficient is profoundly influenced by the size, shape and arrangement of the molecules (5).

Although the world's first viscometer is found as a temperature-compensated water clock that dates back to 2500 B.C., the first formal attempt at analysis was performed by Isaac Newton in the late 1600s (40). He defined viscosity by considering the following model. Two parallel planes of liquid of area, A, are separated by a distance, dx, and move at a velocity differential, dv. Newton assumed that the force, F, required to maintain this difference in speed was proportional to the velocity gradient, dv/dx (γ), through the material (3).

Newton developed the equation:

$$\tau = \frac{F}{A} = \mu(\frac{dv}{dx}) \qquad (4)$$

or

$$\mu = \tau / \frac{dv}{dx} \qquad (5)$$

μ is the coefficient of viscosity and is defined as the ratio of shear stress to the strain rate, γ .

A material requiring a shear stress of 1 dyne/cm^2 to produce a strain rate of one inverse second has a viscosity of one poise or 100 centipoise. It is only by coincidence that water at room temperature has a viscosity of one centipoise (24).

Newtonian fluids are defined to be those exhibiting a direct proportionality between shear stress and strain rate (34). In other words ratio of strain rate to shear stress is constant as shown in Figure 10.

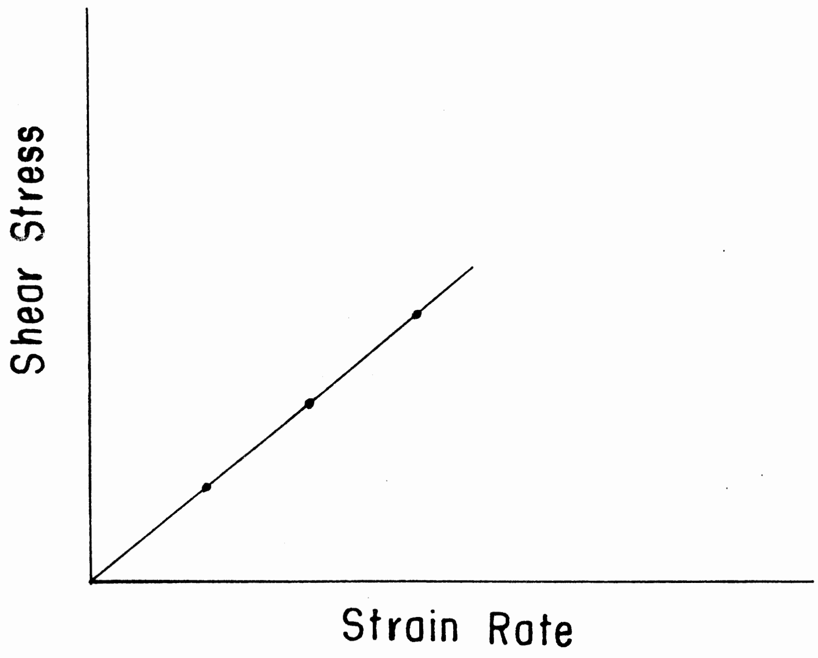

Figure 10. Newtonian Fluids

All those fluids for which the flow curve (shear stress versus strain rate) is not linear through the origin at a given temperature and pressure are said to be non-Newtonian (35).

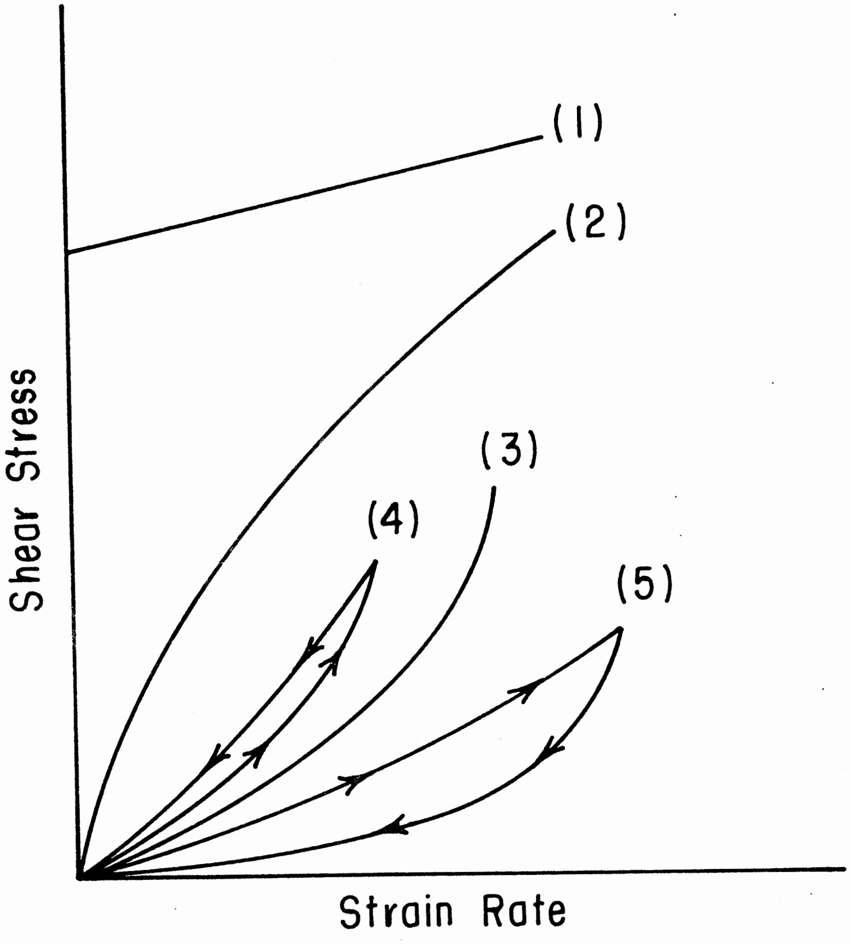

Figure 11. Typical of Flow Curves for Non-Newtonian Fluids. Curves 1 Through 5 Represent Typical Behaviour of True Plastic, Pseudoplastic, Dilatant, Thixotropic and Rheopectic Fluids Respectively

Non-Newtonian fluids are classified according to the general pattern of functional dependence of strain rate on shear stress (and in some cases time, and occasionally frequency of vibrational agitation) (42). These may be placed in one of six general groups: true plastic, pseudo plastic, dilatant, thixotropic, rheopectic, or viscoelastic (Figure 11). If the shear stress-strain rate relationship of non-Newtonian fluid plots as a straight line in log-log coordinates, the fluid is classified as "Power Law Fluid" (35) and is defined by equation 6.

$$\tau = K\left(\frac{dv}{dx}\right)^n \qquad (6)$$

where: τ = Shear stress, $\frac{\text{dyne}}{\text{cm}^2}$

K = Viscometric index, $\frac{\text{dyne-sec}^n}{\text{cm}^2}$

n = Viscometric coefficient, dimensionless

$\frac{dv}{dx}$ = Strain rate, sec^{-1}

Rotational viscometers are widely used for the study of flow properties of non-newtonian materials. A great number of different designs of rotational viscometer have been described in the literature. Essentially a rotational viscometer comprises two members, separated by the material under test, which are able to rotate relative to one another about a common axis of symmetry. As one member rotates, the other tends to be dragged by it, i.e. the test material transmits a torque to the second member. In all rotational viscometers it is the relationship between torque and angular velocity that is used to characterize fluid properties of the test material (5).

CHAPTER III

MATERIAL, EQUIPMENT AND METHOD

Ground Corn

Whole corn was purchased locally and ground in a Wiley's Laboratory mill #4 using three screen sizes 3, 4 and 5mm. Sieve analysis of data from each screen are shown in Appendix A and plotted in Figure 12. For each sample, geometric mean diameter and standard deviation were calculated by equations 7 and 8 (20). The results are shown in Figure 12.

$$d_{gm} = \log^{-1}\left[\sum(W_i \log d_i)/\sum W_i\right] \tag{7}$$

$$SD_{gm} = \log^{-1}\left[\frac{\sum W_i(\log d_i - \log d_{gm})^2}{\sum W_i}\right]^{1/2} \tag{8}$$

where d_i = diameter of sieve openings of i'th sieve, micron

d_{i+1} = diameter of openings in the next lower than i'th sieve (just above in a set), micron

d_{gm} = geometric mean diameter, micron

d_i = geometric mean diameter of particles on i'th sieve = $[(d_i)(d_{i+1})]^{1/2}$

SD_{gm} = Standard deviation

W_i = weight fraction on i'th sieve

Starch content of corn normally varies between 69% and

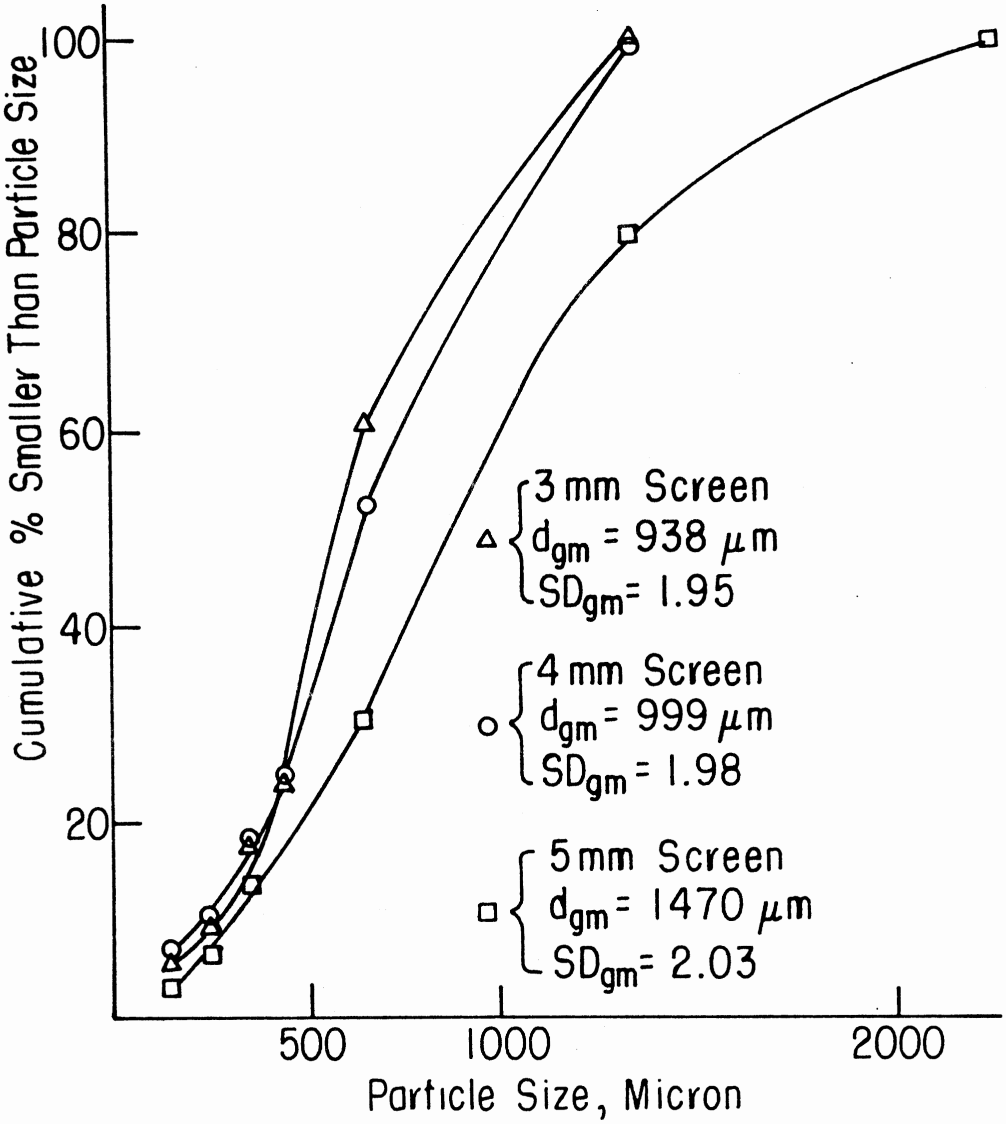

Figure 12. Size Distribution of Ground Corn as a Function of Screen Size; d_{gm} = Geometric Mean Diameter; SD_{gm} = Geometric Standard Deviation

73% (35,6). During cooking of corn in a water slurry, water is bound to the starch molecules resulting in large changes in viscometric properties of the slurry.

Enzyme

It is widely believed that α-amylases from a number of sources hydrolyze starch at random points in the polymer chain to give a random distribution of products that eventually become identical (28). α-Amylases cause hydrolysis of the interior $(1 \rightarrow 4)$-α-D glucosidic bonds of amylose and amylopectin, and is thus an endo-hydrolase. The bond rupture can occur almost anywhere in a chain of α-D$(1 \rightarrow 4)$-linked D-glucosyl residues, so long as there are at least 6 D-glucosyl residues on one side, and at least 3 on the other side of the bond to be broken (18). Consequently the viscosity of gelatinous starch mashes is reduced. The final products of digestion of starch by α-amylases are soluble dextrin, glucose and maltose (18).

TAKA-THERM, a liquid bacterial alpha-amylase of Bacillus lichentormis var. origin, produced by Miles Laboratories, Inc. was used in this experiment. TAKA-THERM exhibits exceptional thermostability and can liquify starches at temperatures above 90°C (194°F) (21).

Equipment and Usage

Cooking Mechanism

Ground corn was cooked in a 15 liter glass jar covered

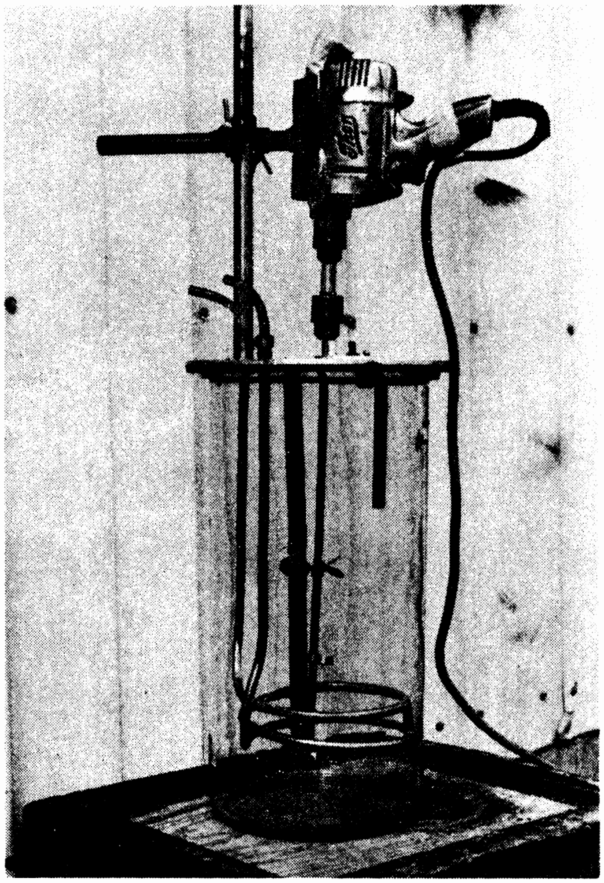

Figure 13. The Apparatus Used Used for Cooking of Corn-Water Slurry for Ethanol Production

with a cap (Figure 13). The agitator (Figure 13) was mounted on a steel rod (Figure 13) which was fastened to the cap. An extension rod (Figure 13) with three agitation blades (Figure 13) was chucked to the drill, passing through a hole mounted at the center of the cap.

To cook the corn, steam was passed through a copper heat exchanger (Figure 13). Heating rate was controlled by controlling steam flow rate through the heat exchanger. Copper-constantan thermocouples were used to monitor temperature of the slurry.

Viscometer

The Brookfield Synchro-Lectric viscometer model RVT with seven RV spindles and a modified LV-2 cylindrical spindle, equipped with Helipath stand (Fig. 14) was used to measure viscosity during cooking. The viscometer consisted of a synchronous motor, a calibrated torsional spring, a dial for reading angular displacement of the spring and a series of spindles of various sizes. The model RVT was calibrated for angular velocities of 0.5, 1, 2.5, 5, 10, 20, 50 and 100 rev/min. The viscosity measuring range was controlled by selection of appropriate spindle. The spindle was driven by the motor through the torsional spring. The spring measured the viscous drag as the cylinder rotated in a stationary container. The full scale torque of the model RVT torsional spring was 0.7187×10^{-3} Nt.m. The cylindrical spindle used in this experiment has a 5.128 mm

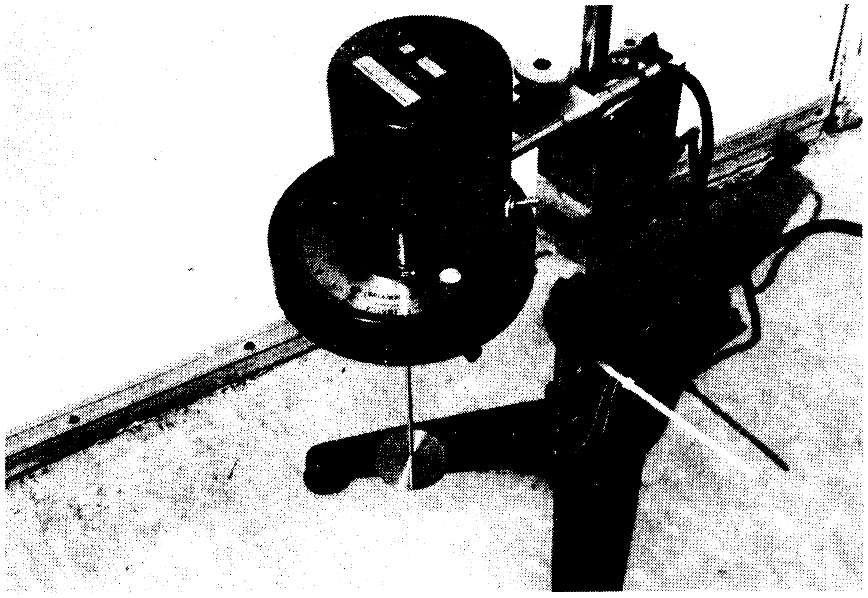

Figure 14. Brookfield's Synchrometric Viscometer Shown with RV#2 Spindle

diameter. A standard 600 mm Griffin beaker was used for measuring viscosity of the liquid. Van Wazer et al. (41) developed the following formulas for determining shear stress and strain rate.

$$\tau = \frac{\theta K_1}{2\pi R_s L} \tag{9}$$

$$\gamma = \frac{2\omega R_c R_s}{d^2 (R_c - R_s)} \tag{10}$$

$$\bar{\gamma} = \frac{2\omega R_c R_s}{R_c - R_s} \tag{11}$$

where τ = shear stress, dyne/cm^2

γ = strain rate, sec^{-1}

$\bar{\gamma}$ = average strain rate, sec^{-1}

θ = dial reading, dimensionless

ω = Angular velocity of the spindle, radians/sec

K_1 = Constant for torsional spring, dyne-cm

R_s = Radius of spindle, .5128 cm

R_c = Radius of container, 8.3 cm

L = Spindle length plus correction for end effect for RV #2 cylindrical spindle: 6.121 cm.

d = distance from the axis of rotation at which strain rate is being calculated, cm

Experimental Design

Viscometric properties of corn-water slurries during cooking were determined for three different sizes of ground corn and three levels of mass fraction of corn to the total slurry mass. Ground corn passing through 3, 4 and 5 mm screen on Wiley's laboratory mill were used. Mass distributions for sieve analysis for the three sizes are shown in Figure 12. Mass fractions chosen were 1.787, 2.233, and 2.680 lit/kg as shown in Tables I and II. The experiments were conducted in a completely random design with three replications.

For a typical days run, ground corn, water, and α-amylase enzyme (TAKA-THERM) were measured according to Table I and II. Heat and agitation was applied to the slurry and viscometric properties of the corn-water slurry was measured at constant temperature intervals beginning at 165°F and ending at 200°F. Vigorous agitation of the slurry was maintained throughout the cooking process. Figures 15 and 16 show agitation of slurry before and during cooking.

TABLE I

EXPERIMENTAL PLAN FOR DETERMINING APPARENT VISCOSITY OF CORN-WATER SLURRIES AT 150°F-200°F

Mass Fraction lit/kg	Screen Size mm	Corn Mass kg	Water Volume lit	TAKA-THERM gm
1.787	3	4.47	8	9.84
	4	4.47	8	9.84
	5	4.47	8	9.84
2.233	3	4.09	9	9
	4	4.09	9	9
	5	4.09	9	9
2.680	3	3.74	10	8.23
	4	3.74	10	8.23
	5	3.74	10	8.23

TABLE II

EXPERIMENTAL PLAN FOR DETERMING SHEAR STRESS-STRAIN RATE RELATIONSHIP OF CORN-WATER SLURRY AT 165°F AND 200°F

Mass Fraction lit/kg	Screen Size mm	Corn Mass kg	Water Volume lit	TAKA-THERM gm
1.787	4	2.24	4	4.92
2.233	4	2.05	4.5	4.5
2.680	4	1.87	5	4.12

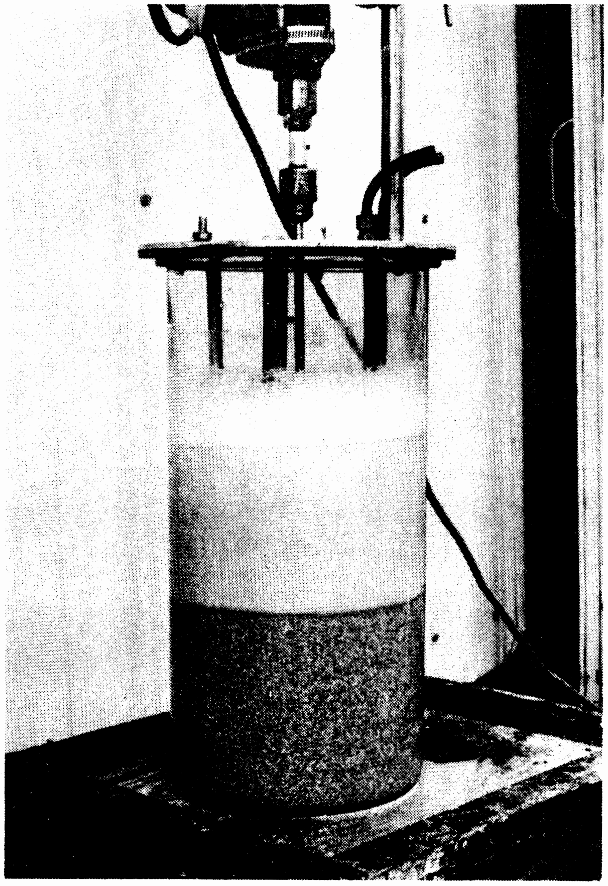

Figure 15. Separation of Corn and Water Shown Before Operation of Agitation System

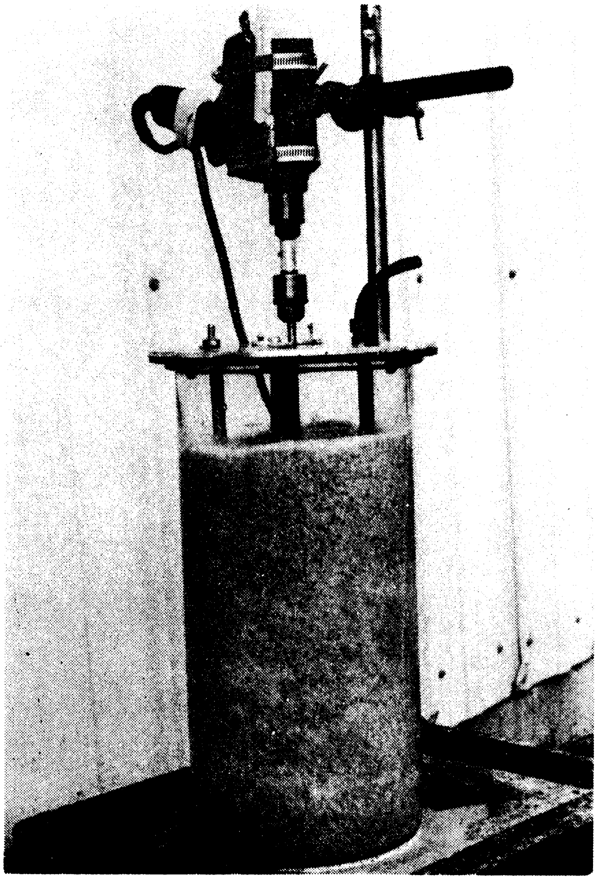

Figure 16. Cooking Vessel in Operation Showing Vigorous Agitation Maintained Throughout Viscometric Tests

CHAPTER IV

RESULTS AND DISCUSSION

Viscometric History

Apparent viscosities of ground corn slurries at 150°F-200°F (65.5-93.3°C) are tabulated in Appendix B, and plotted in Appendix C. The effect of particle size on apparent viscosity for different mass fractions of ground corn are shown in Figures 17-19. For mass fraction of 1.787 lit/kg, the apparent viscosity of 3mm corn mash is higher than those for corn ground on 4 and 5 mm screens (Figure 17). As shown in Figure 18, for mass fraction of 2.233 lit/kg, the apparent viscosity of 3mm corn mash is unexpectedly lower than those for 4 or 5mm. As expected the value of 5mm is lower than 4mm. It is apparent from Figure 19 that for a mass fraction of 2.680 lit/kg, increasing the ground corn particle size doesn't affect apparent viscosity of the mash. This is due to the effect of higher percentage of available starch molecules which are obtained at smaller particle size of ground corn. During slurry heating, the starch molecule increase in volume due to rapid absorption of water. For mass fraction of 1.787 lit/kg, the starch granules absorb most of the water but the enzyme can not disintegrate them completely since they remain in a

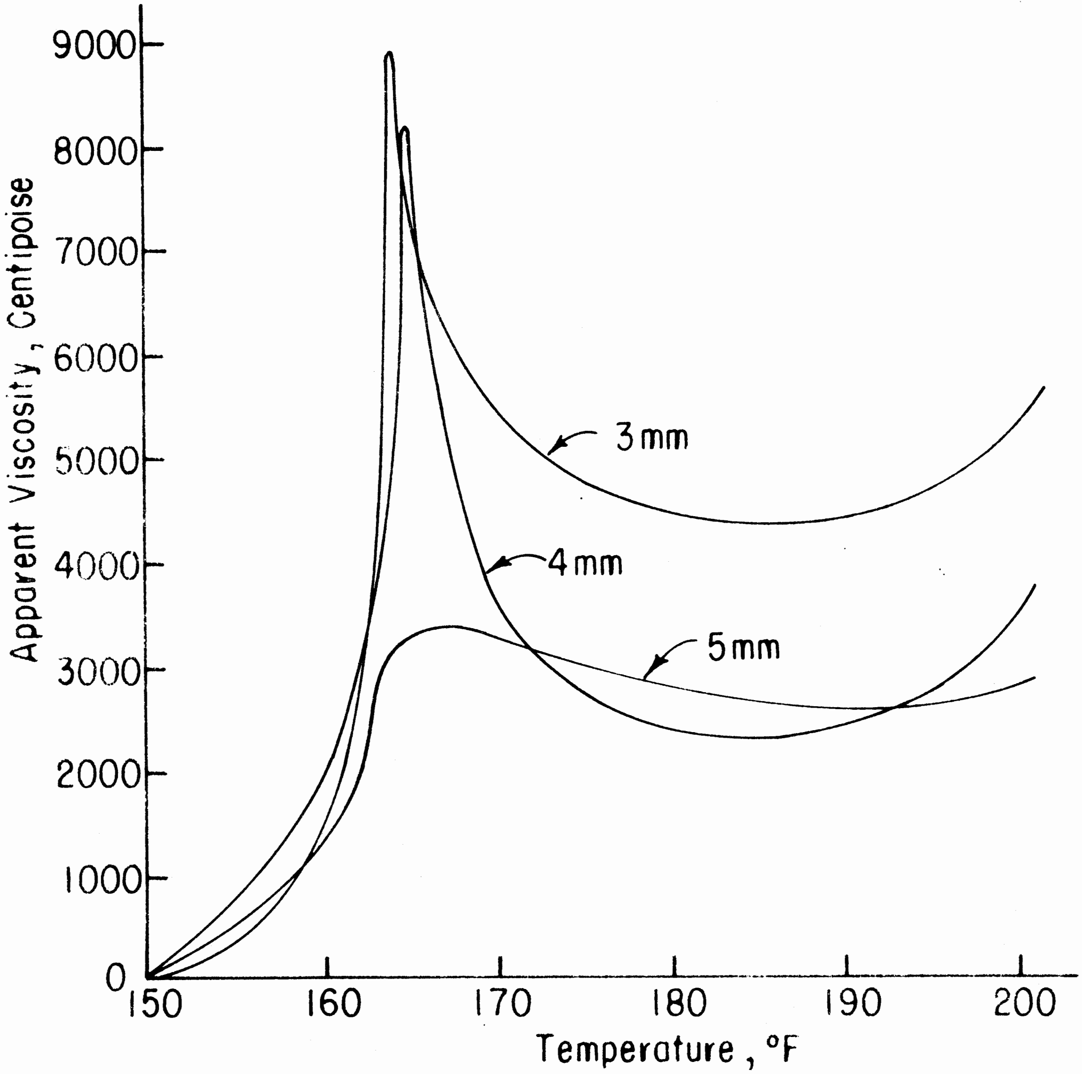

Figure 17. Effect of Ground Corn Particle Size on Apparent Viscosity of Slurry During Cooking at One Atmosphere. The Solid Mass Fraction is 1.787 lit/kg

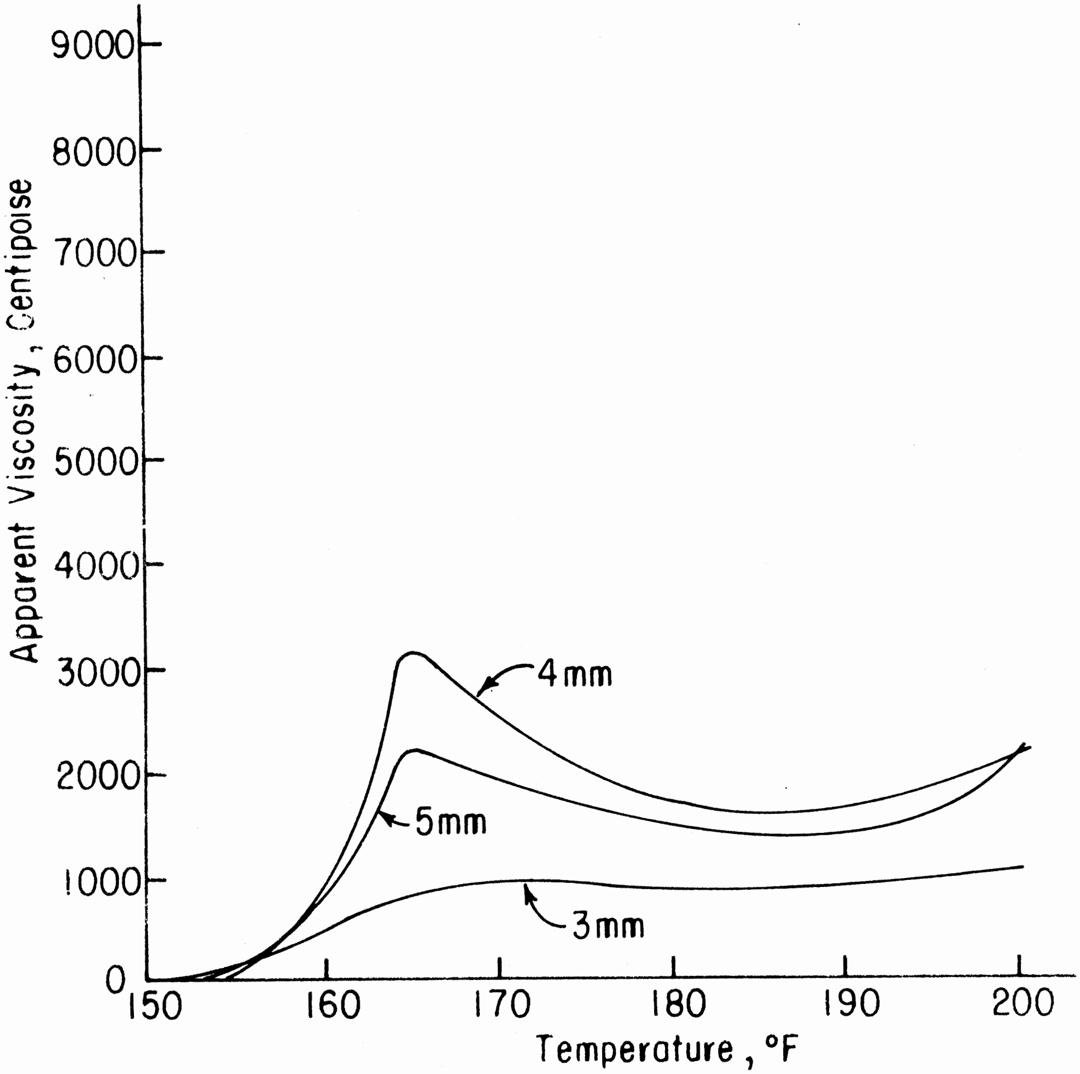

Figure 18. Effect of Ground Corn Particle Size on Apparent Viscosity of Slurry During Cooking at One Atmosphere. The Solid Mass Fraction is 2.233 lit/kg

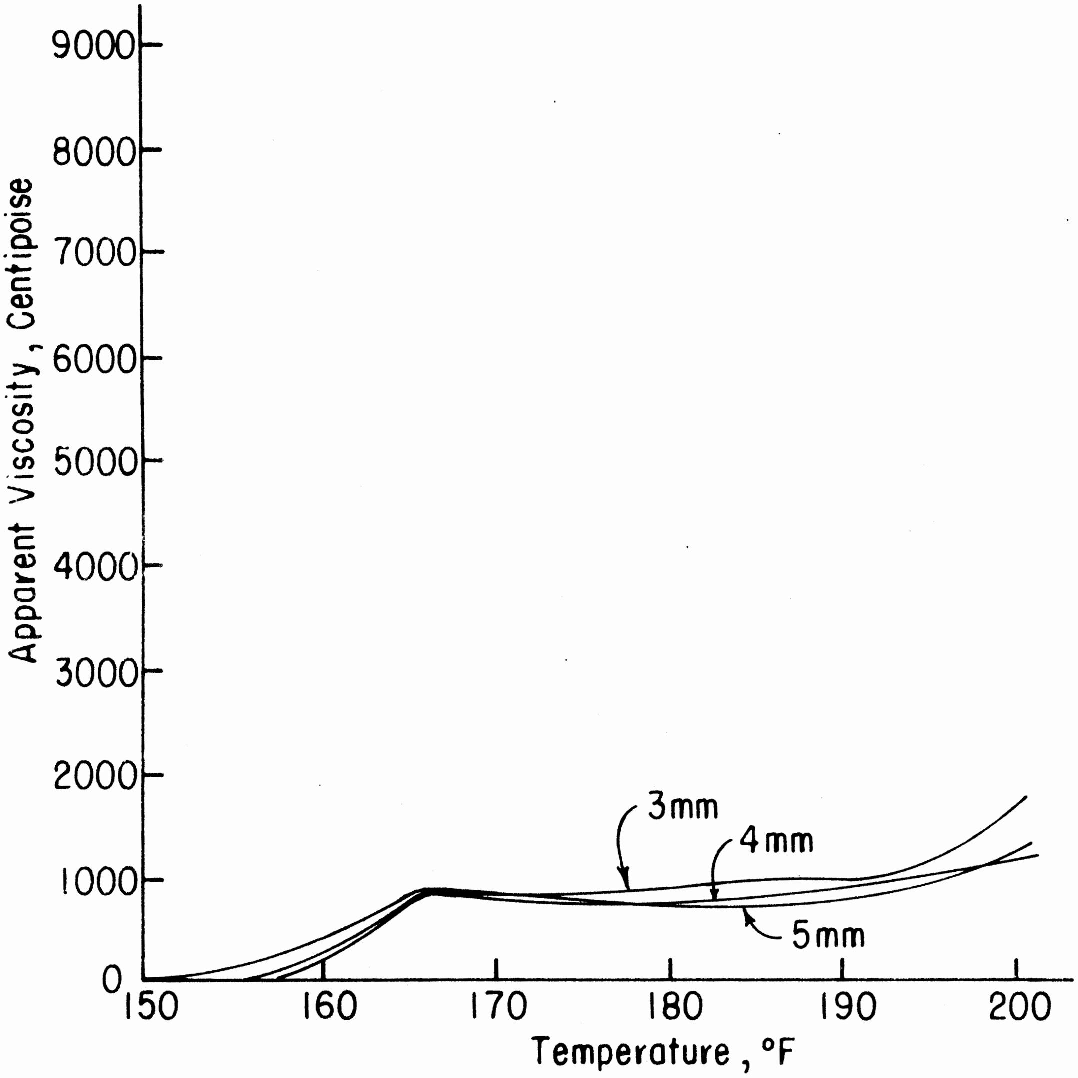

Figure 19. Effect of Ground Corn Particle Size on Apparent Viscosity of Slurry During Cook-in at One Atmosphere. The Solid Mass Fraction is 2.680 lit/kg

partially crystaline form. However since higher percentage of starch molecules are available at smaller particle size, higher apparent viscosity of mash is observed. For a mass fraction of 2.233 lit/kg this phenomena still exists with the exception of the 3mm ground corn particle size in which the mash show a lower apparent viscosity than those for 4 and 5mm. For the grain ground on the 3mm screen the amount of water and enzyme is optimum for the interaction, therefore the starch molecule is broken down to dextrin easily, resulting in lower viscosity. The lower percentage of free starch molecules should make the apparent viscosity of corn mash ground through 4 and 5mm screens less than the apparent viscosity of corn mash passing a 3mm screen. However the starch molecules in large particle sizes still hydrate but not as much as smaller particle sizes, and so the suspended particles make the viscous fluids thicker than expected. For a mass fraction of 2.680 lit/kg, there is sufficient water to allow the alpha-amylase enzyme to convert the free starch molecules to dextrins and the effect of suspended particles on apparent viscosity in this mass fraction is negligible.

The apparent viscosity of mash for 1.787 lit/kg is generally higher than 2.233, and 2.680 lit/kg respectively as shown in Figures 20-22. Mazurs et al. (17) showed similar results with acid modified starch. This occurs because the granule will first swell and then dissolve to give a molecularly dispersed solution. At high concentrations,

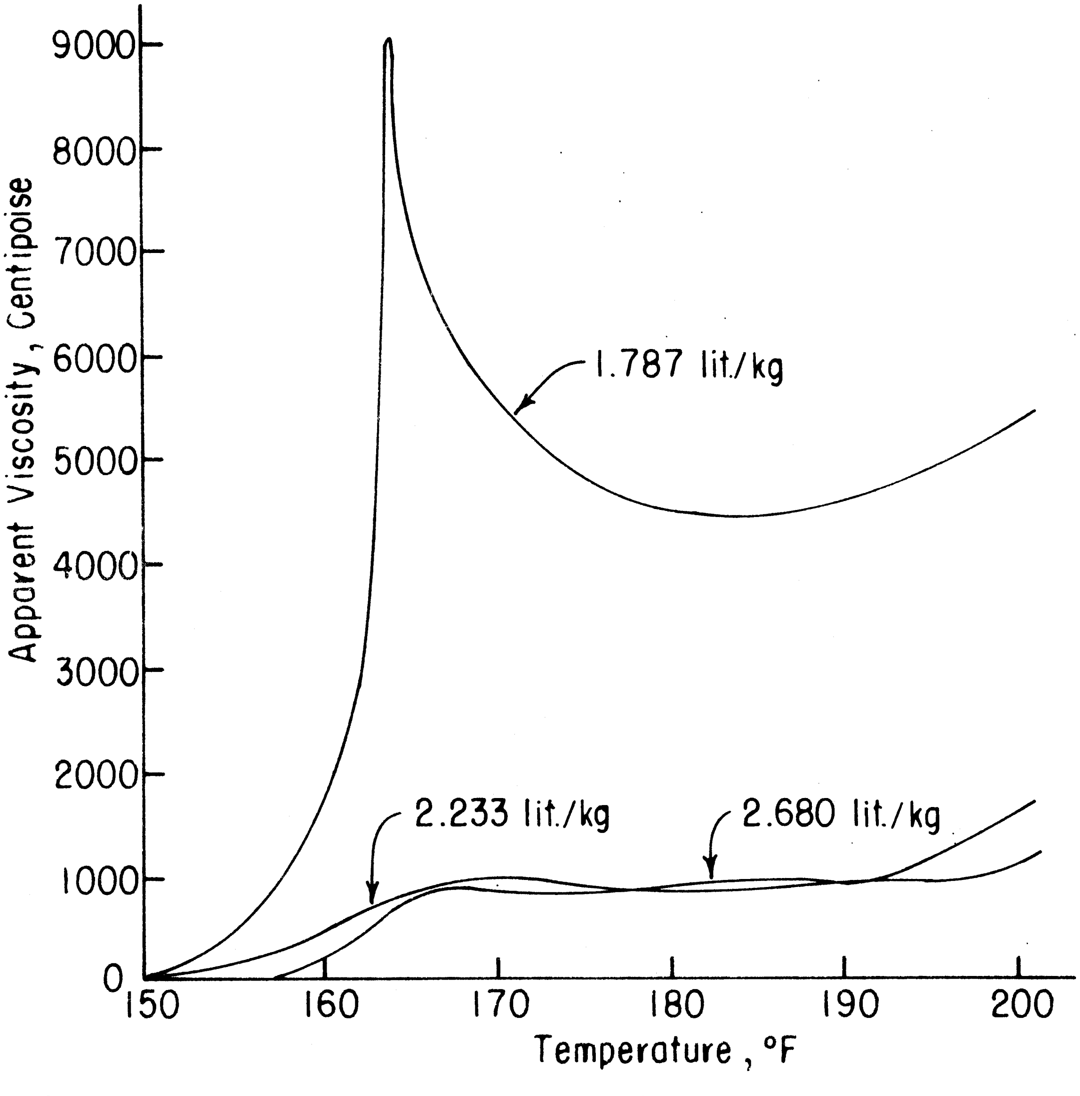

Figure 20. Effect of Mass Fraction on Apparent Viscosity of Mash During Cooking at One Atmosphere for 3mm Ground Corn Particle Size

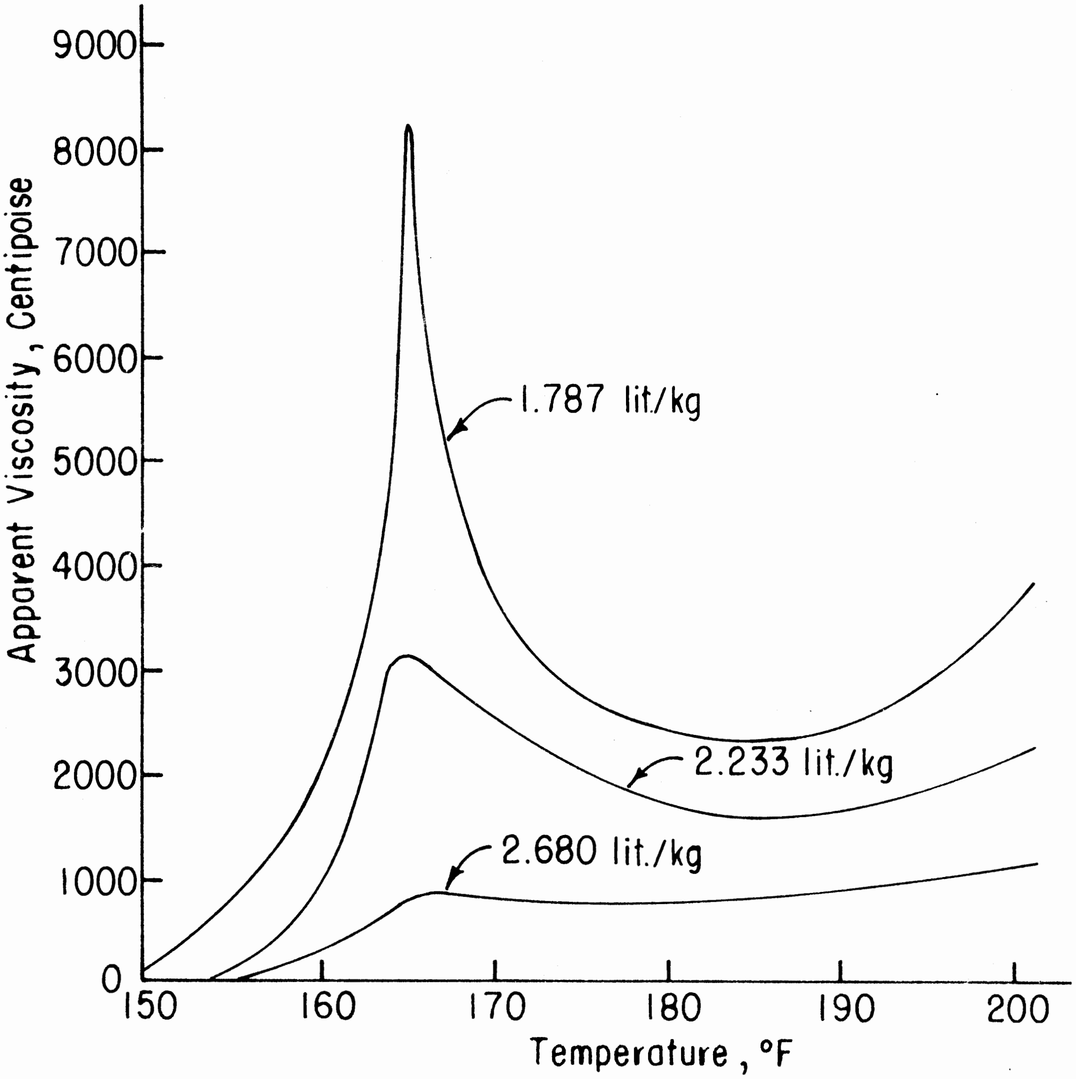

Figure 21. Effect of Mass Fraction on Apparent Viscosity of Mash During Cooking at One Atmosphere for 4mm Ground Corn Particle Size

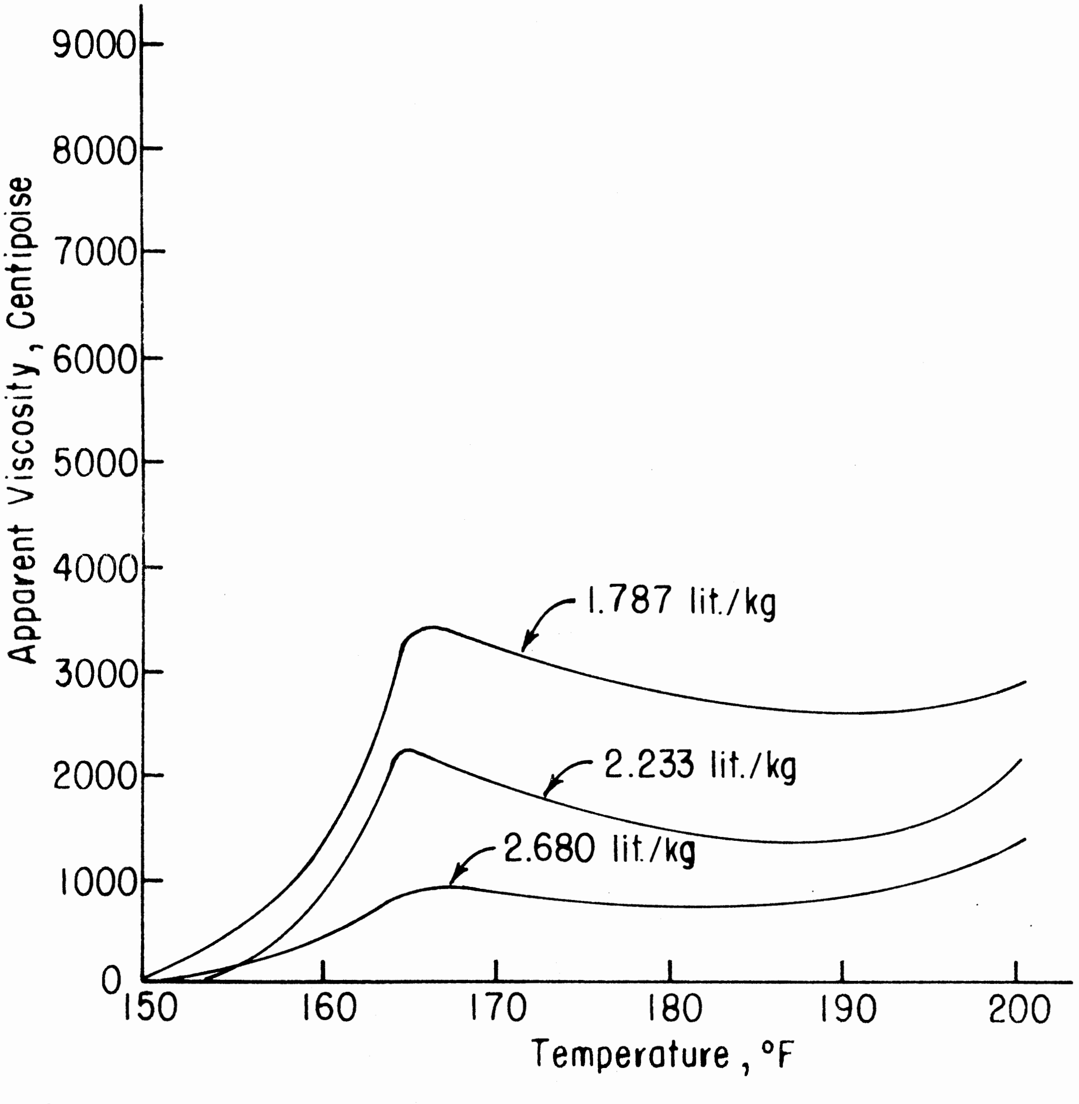

Figure 22. Effect of Mass Fraction on Apparent Viscosity of Mash During Cooking at One Atmosphere for 5mm Ground Corn Particle Size

the latter is unstable and tends to set up a gel. Continual mechanical agitation breaks down this gel structure with consequent thinning of the paste (17). Enzymatic effect is similar to acid modification of corn starch, since it also breaks down the starch molecule.

Peak Apparent Viscosity

The highest apparent viscosity occurred at 165°C-170°F (74.4-76.7°C). These values are tabulated in Table III. An analysis of variance was performed on the data to test the statistical significance of variation due to particle size and mass fraction. The analysis is shown in Table IV. The results indicate significant differences in mean value of peak apparent viscosity at different levels of mass fraction ($\alpha = .0001$). This confirms work of previous investigators (9, 23) showing that apparent viscosity is a function of mass fraction. Particle size effect ($\alpha = .05$) and interaction between particle size and mass fraction ($\alpha = .0075$) are also significant.

Duncan's multiple range test was performed to compare each treatment mean with other treatment means as shown in Tables V and VI.

The results in Table V and Figure 23 show significant differences on peak apparent viscosity due to mass fraction. The effect of particle size on apparent viscosity (Figure 24) is misleading since these values include the peak apparent viscosity of mash at mass fraction of 2.233

TABLE III

MAXIMUM APPARENT VISCOSITY AT FIRST PEAK, μ_{maxP1} AND AT 200°F, μ_{maxP2}, AS OBTAINED IN APPENDIX B

Repl.	Screen Size mm	Mass Fraction lit/kg	Peak Apparent Viscosity centipoise	Apparent Viscosity at 200°F centipoise
1	3	1.787	6340	6435
2	3	1.787	9100	3800
3	3	1.787	7645	6670
1	3	2.233	860	800
2	3	2.233	1100	1060
3	3	2.233	1020	880
1	3	2.680	1160	1220
2	3	2.680	820	1380
3	3	2.680	720	1760
1	4	1.787	2710	6725
2	4	1.787	8220	3830
3	4	1.787	8220	3350
1	4	2.233	3000	2550
2	4	2.233	4970	1870
3	4	2.233	3200	1725
1	4	2.680	1000	1500
2	4	2.680	840	860
3	4	2.680	820	820

TABLE III (Continued)

Repl.	Sreen Size mm	Mass Fraction lit/kg	Peak Apparent Viscosity centipoise	Apparent Viscosity at 200°F centipoise
1	5	1.787	4400	7000
2	5	1.787	1970	2710
3	5	1.787	3050	3830
1	5	2.233	2300	2600
2	5	2.233	2600	1970
3	5	2.233	2020	1820
1	5	2.680	640	1700
2	5	2.680	900	1340
3	5	2.680	950	1400

TABLE IV

ANALYSIS OF VARIANCE OF THE PEAK APPARENT VISCOSITY FOR GROUND CORN SIZE AND MASS FRACTION

Source	Degree of freedom	Sum of Squares	F Ratio	Significance Level*
Corrected total	26	186117035		
Size	2	11729368	3.55	0.0502
Mass Fraction	2	112188468	33.94	0.0001
Size * Mass Fraction	4	32451314	4.91	0.0075
Error	18	29747883		

*Probability of error in rejecting a null hypothesis of significance of the source of variation.

lit/kg which is lower than expected (the reason has been explained in the "viscometric history" section).

Apparent Viscosity at 200°F

Apparent viscosity was determined at 200°F because it is typical of maximum cooking temperature at one atmosphere. These values are shown in Table III. An analysis of variance shown in Table VII tests the statistical significance of variation due to particle size and mass fraction on apparent viscosity at 200°F. Effect of mass fraction on apparent viscosity of mash at 200°F was significant

TABLE V

DUNCAN'S MULTIPLE RANGE TEST FOR PEAK APPARENT VISCOSITY

Grouping	Mean (centipoise)	N	Size (mm)
B	3196.1	9	3
A	3664.4	9	4
B	2092.2	9	5

Means with the same letter (as in "grouping" column are not significantly different at the 95 percent confidence level (α = .05)

TABLE VI

DUNCAN'S MULTIPLE RANGE TEST FOR PEAK APPARENT VISCOSITY

Grouping	Mean (centipoise)	N	Mass Fraction (lit/kg)
A	5739.4	9	1.787
B	2341.1	9	2.233
C	872.2	9	2.680

Means with the same letter (as in "grouping" column are not significantly different at the 95 percent confidence level (α = .05)

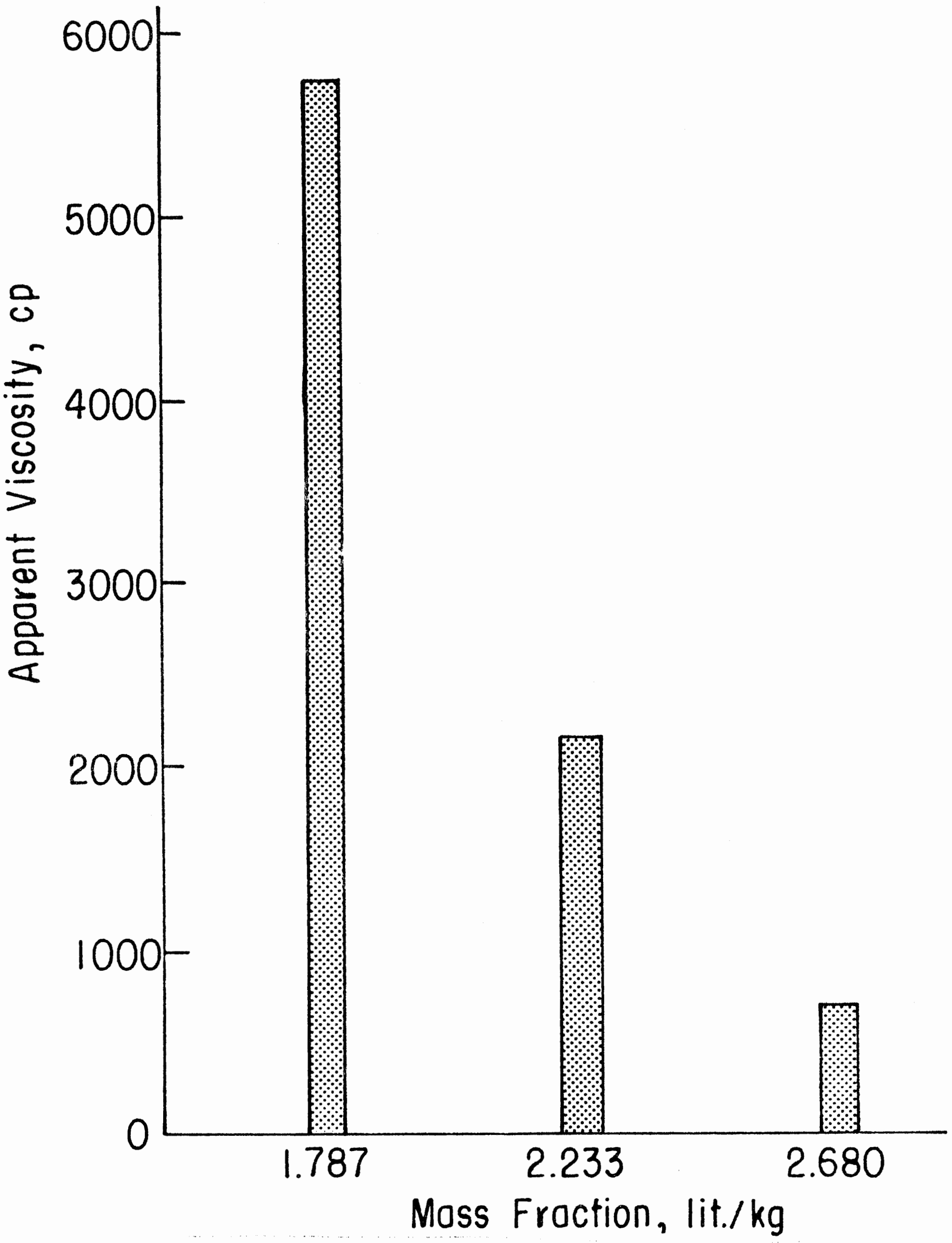

Figure 23. Effect of Mass Fraction on Peak Apparent Viscosity of Corn Mash During Cooking for Ethanol Production

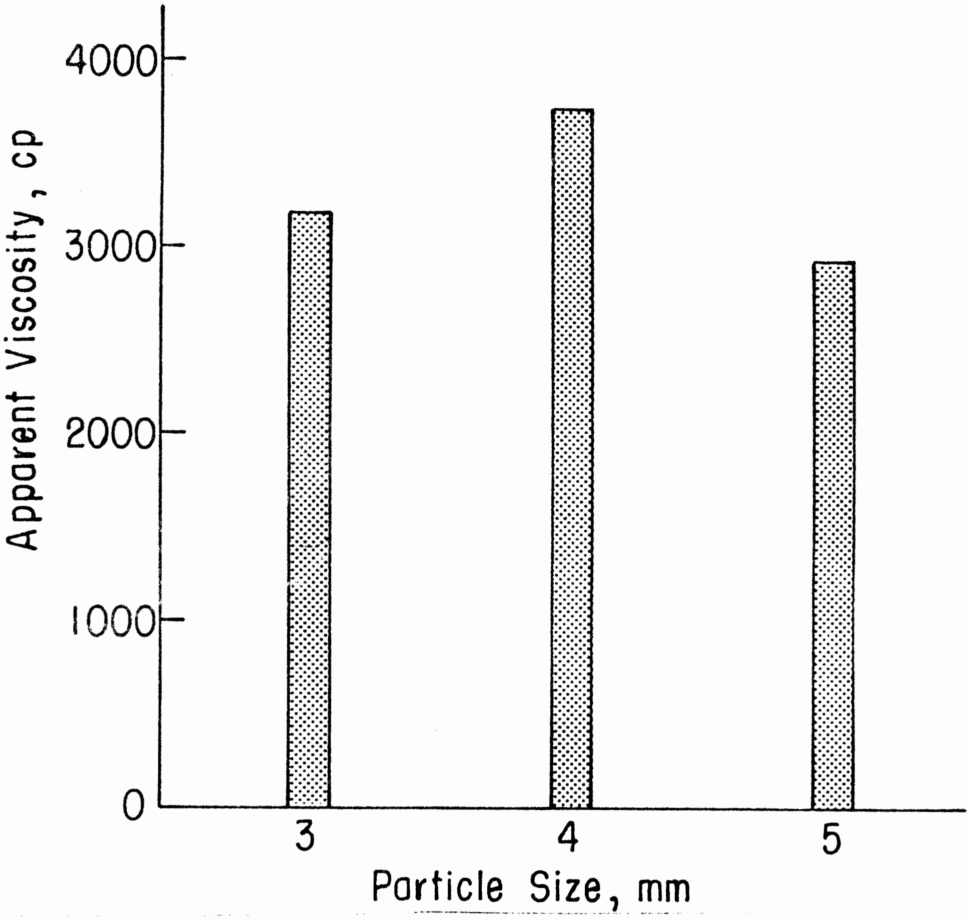

Figure 24. Effect of Ground Corn Particle Size on Peak Apparent Viscosity of Corn Mash During Cooking for Ethanol Production

TABLE VII

ANALYSIS OF VARIANCE OF APPARENT VISCOSITY AT 200°F FOR GROUND CORN SIZE AND MASS FRACTION

Source	Degree of Freedom	Sum of Squares	F ratio	Significance level*
Corrected Total	26	98833363		
Size	2	75313	0.03	.9709
Mass Fraction	2	70519613	27.67	.0001
Size * Mass Fraction	4	5304620	1.04	.4136
Error	18	22933816		

*Probability of error in rejecting a null hypothesis of significance of the source of variation.

(α = .0001).

Duncan's Multiple Range Test was performed to compare each treatment mean with other treatment means and are shown in Tables VIII and IX, and plotted in Figures 25 and 26.

Table VIII and Figure 25 show a significant difference between peak apparent viscosity means at 200°F for mass fractions of 1.787 lit/kg and 2.233 or 2.680 lit/kg. Table IX shows no significant difference between treatment means due to particle size of corn even at the 90 percent confident level (α =.1) (Figure 26).

General Viscometric Relationships

As outlined in the previous chapter, the Brookfield rotary viscometer was used to determine shear stress-strain rate relationships for corn slurries during cooking in the ethyl alcohol process. The LV#2 cylindrical spindle was used and angular displacements were recorded at various angular velocities of the spindle. Shear stress and strain rate were calculated from these data using equations 6 and 7 and are tabulated in Appendix E. Since the results of this study showed that particle size has no significant effect on apparent viscosity of corn-water slurries during cooking, all test for determining shear stress-strain rate relationships were conducted with corn ground using a 4mm screen on the Wiley laboratory mill. The relationships between shear stress and strain rate for the three mass fractions of 1.787, 2.233 and 2.680 lit/kg are plotted in

TABLE VIII

DUNCAN'S MULTIPLE RANGE TEST FOR APPARENT VISCOSITY AT 200°F

Grouping	Mean (Centipoise)	N	Size(mm)
A	2667.2	9	3
A	2581.1	9	4
A	2707.8	9	5

Means with the same letter (as in "grouping" column are not significantly different at the 95 percent confidence level (α = .05)

TABLE IX

DUNCAN'S MULTIPLE RANGE TEST FOR APPARENT VISCOSITY AT 200°F

Grouping	Mean (Centipoise)	N	Mass Fraction (lit/kg)
A	4927.8	9	1.787
B	1697.2	9	2.233
B	1331.1	9	2.680

Means with the same letter (as in "grouping" column) are not significantly different at 95 percent confident level (α = .05)

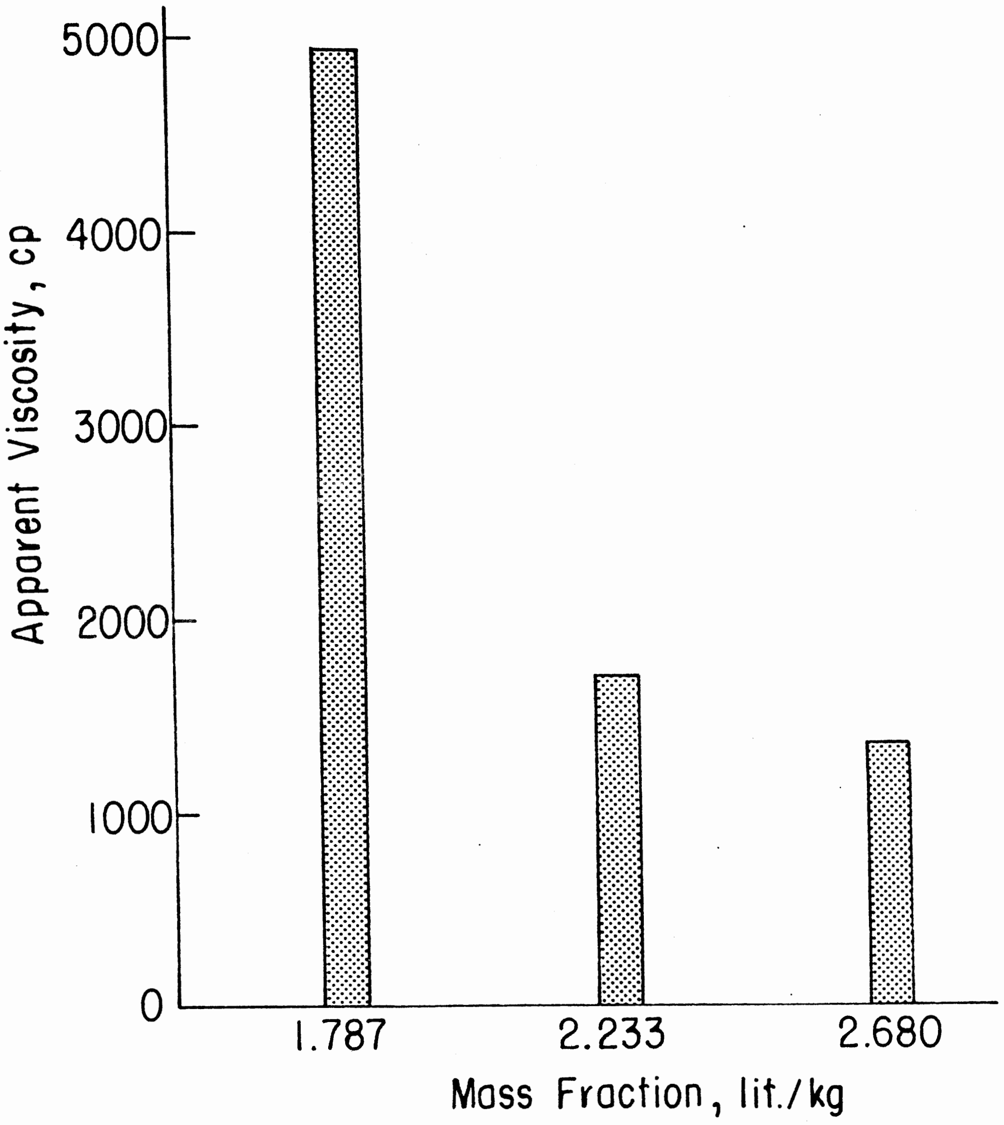

Figure 25. Effect of Mass Fraction on Apparent Viscosity at 200°F of Corn Mash During Cooking for Ethanol Production

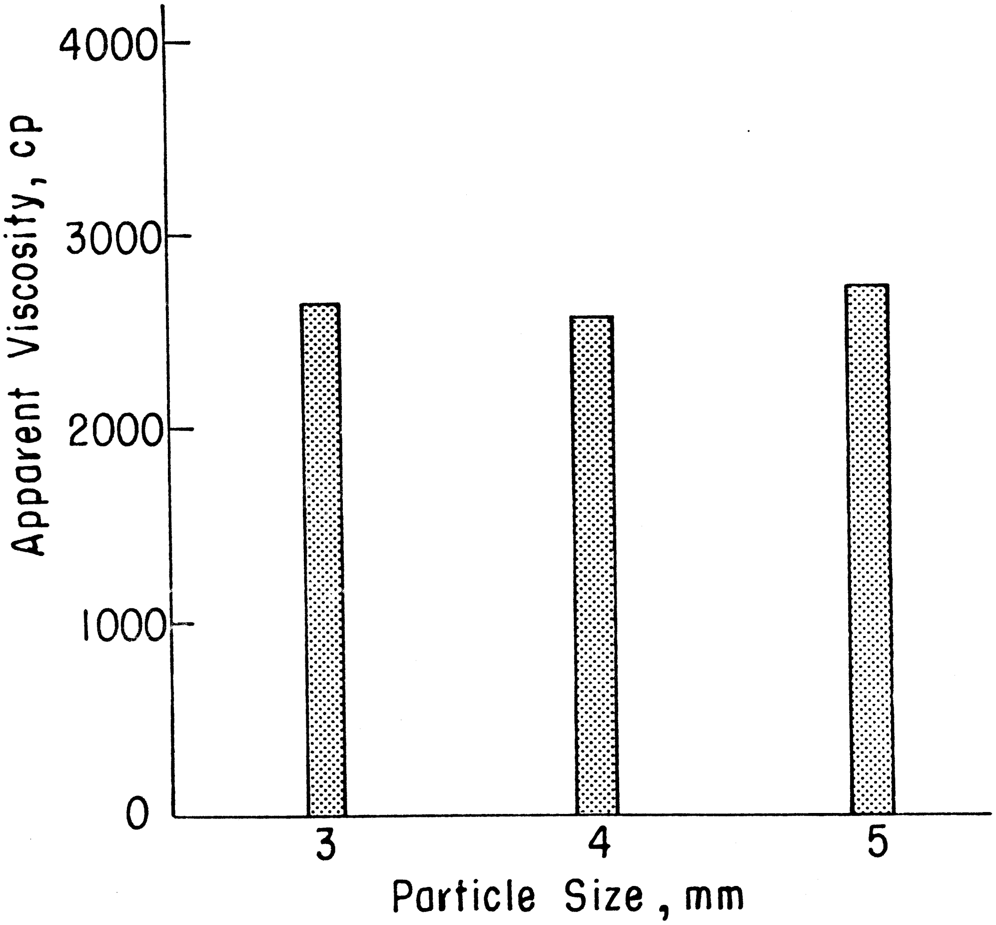

Figure 26. Effect of Ground Corn Particle Size on Apparent Viscosity at 200°F of Corn Mash During Cooking for Ethanol Production

Figures 27-32. The data in all cases plotted as straight lines in log-log coordinates. The corn-water slurries are typical (power law) non-newtonian fluids that obey equation 6:

$$\tau = K(\frac{dv}{dx})^n \qquad (6)$$

where: τ = Shear stress, dyne/cm^2

$\frac{dv}{dx}$ = Strain rate, sec^{-1}

K = Viscometric index, $\frac{\text{dyne-sec}^n}{\text{cm}^2}$

n = Viscometric coefficient, dimensionless

Values for k and n for each experiment are tabulated in Table X with the respective equations shown on each of the Figures 27-32 . The slope of the curves shown in these figures are all greater than unity indicating that corn-water slurries at 165°F and 200° are classified as dilatant fluids.

Figures 27-32 and Table X shows that the viscometric parameters k and n are functions of mass fraction and temperature at which viscometric properties are measured during cooking of the corn water slurry. Shear stress-strain rate curves for the three mass fractions and at 165°F are plotted on one set of coordinates in Figure 33. From this figure it is observed that the intercept of the shear stress-strain rate curve at a strain rate value of 1.0 is a function of the mass fraction of corn to total slurry mass. The effect of mass fraction on the viscometric index k is

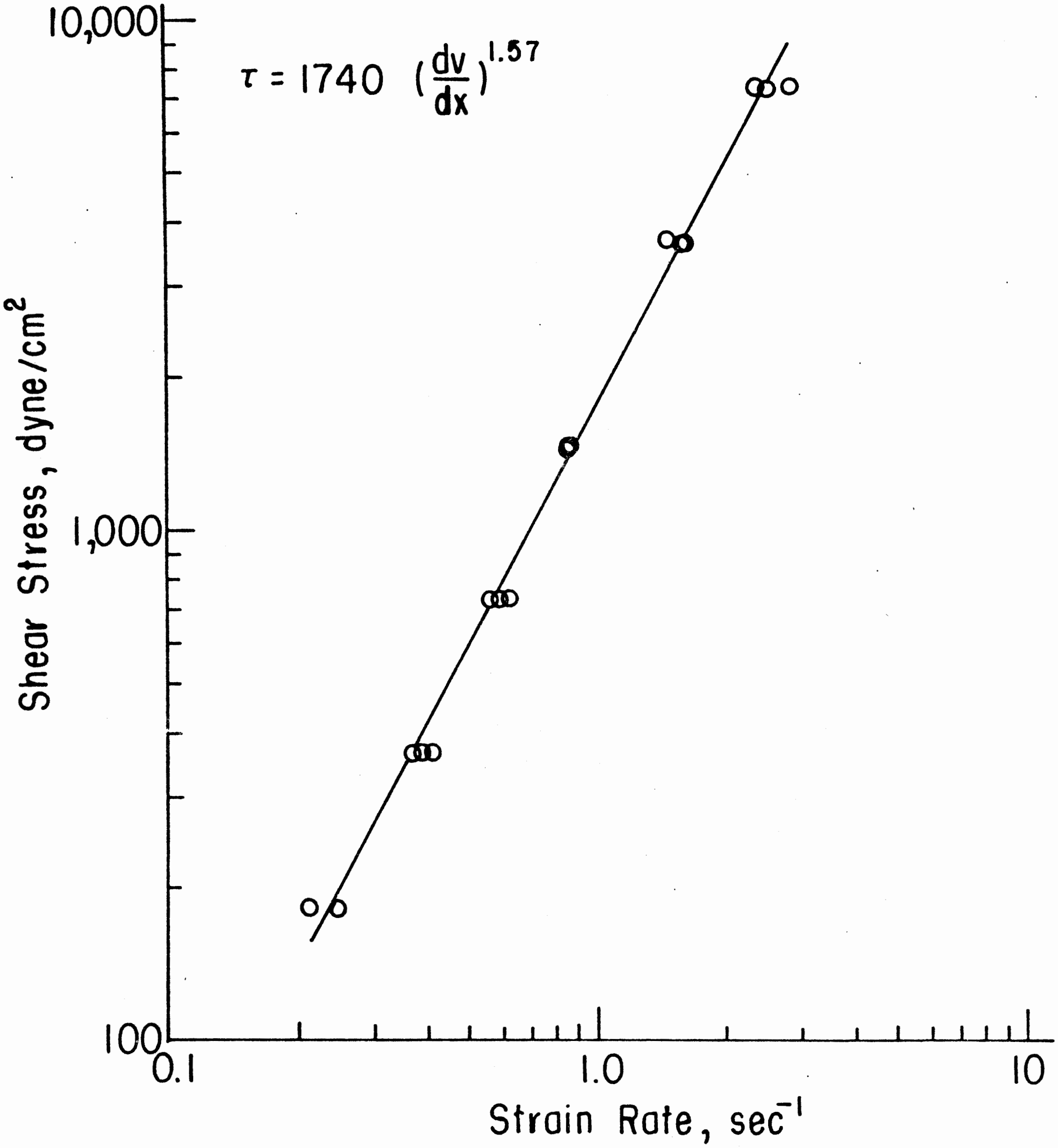

Figure 27. Shear Stress-Strain Rate Relationship for Mass Fraction of 1.787 lit/kg and 4mm Corn Particle Size at 165°F

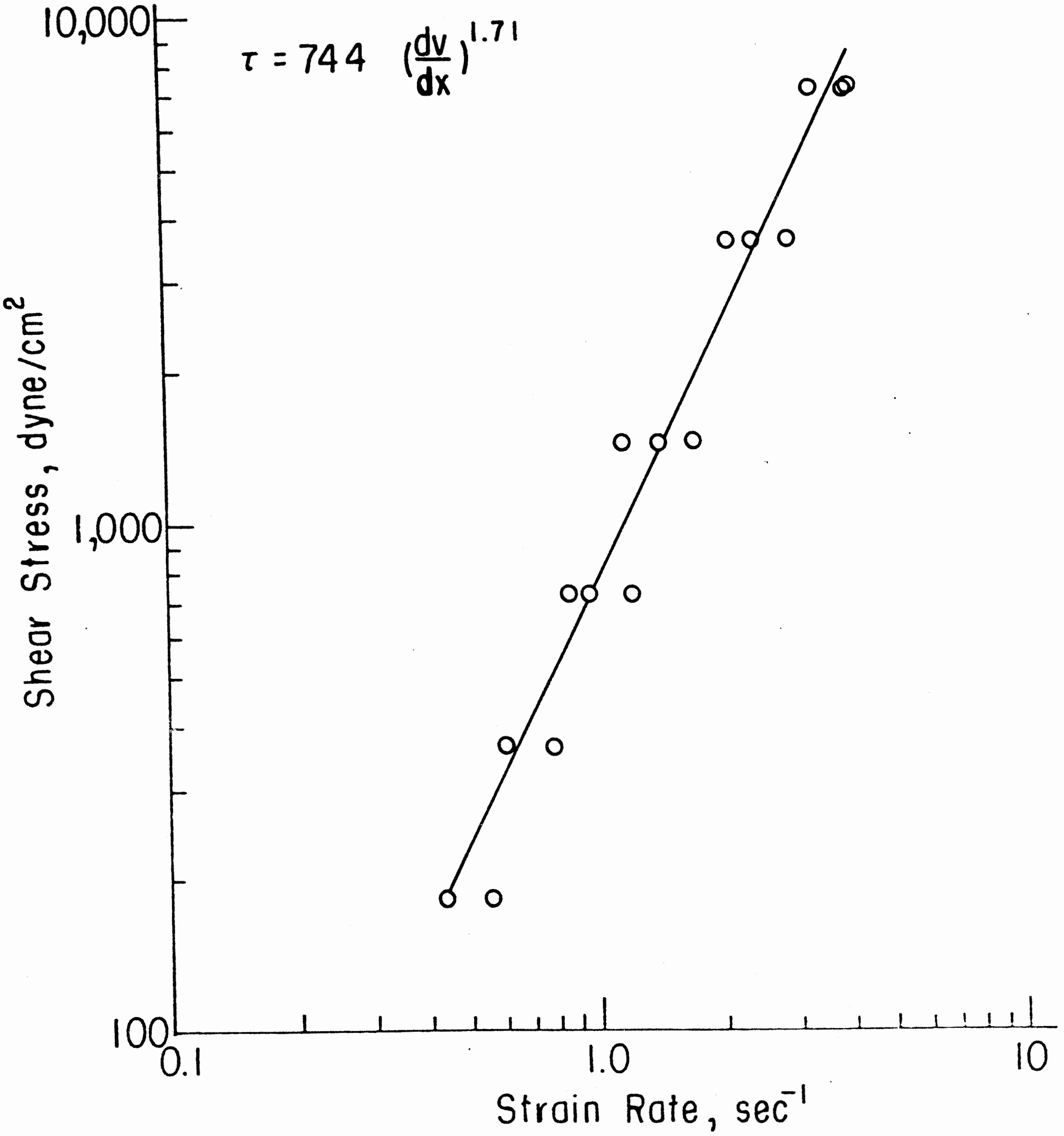

Figure 28. Shear Stress-Strain Rate Relationship for Mass Fraction of 1.787 lit/kg and 4mm Corn Particle Size at 200°F

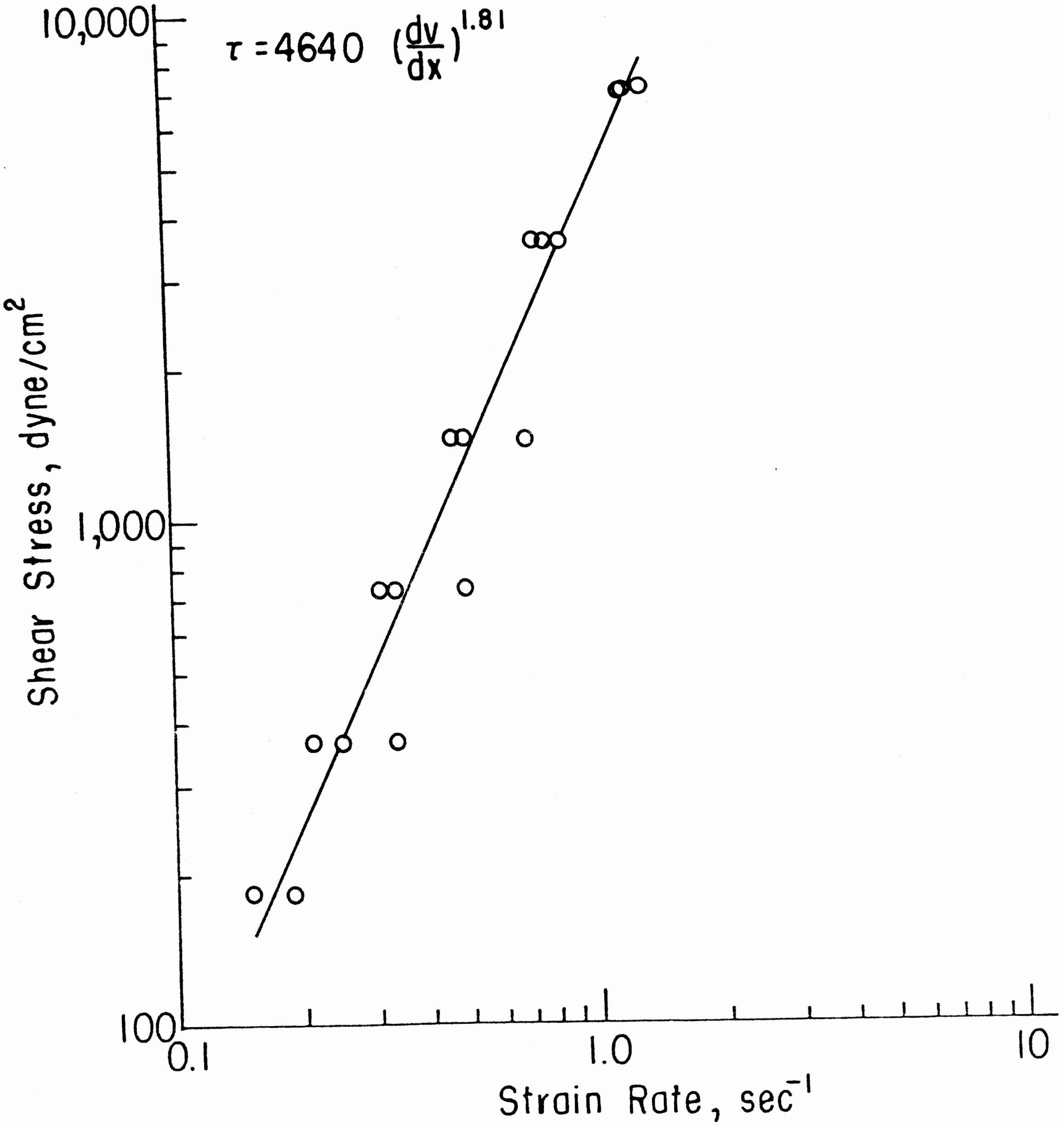

Figure 29. Shear Stress-Strain Rate Relationship for Mass Fraction of 2.233 lit/kg and 4mm Corn Particle Size at 165°F

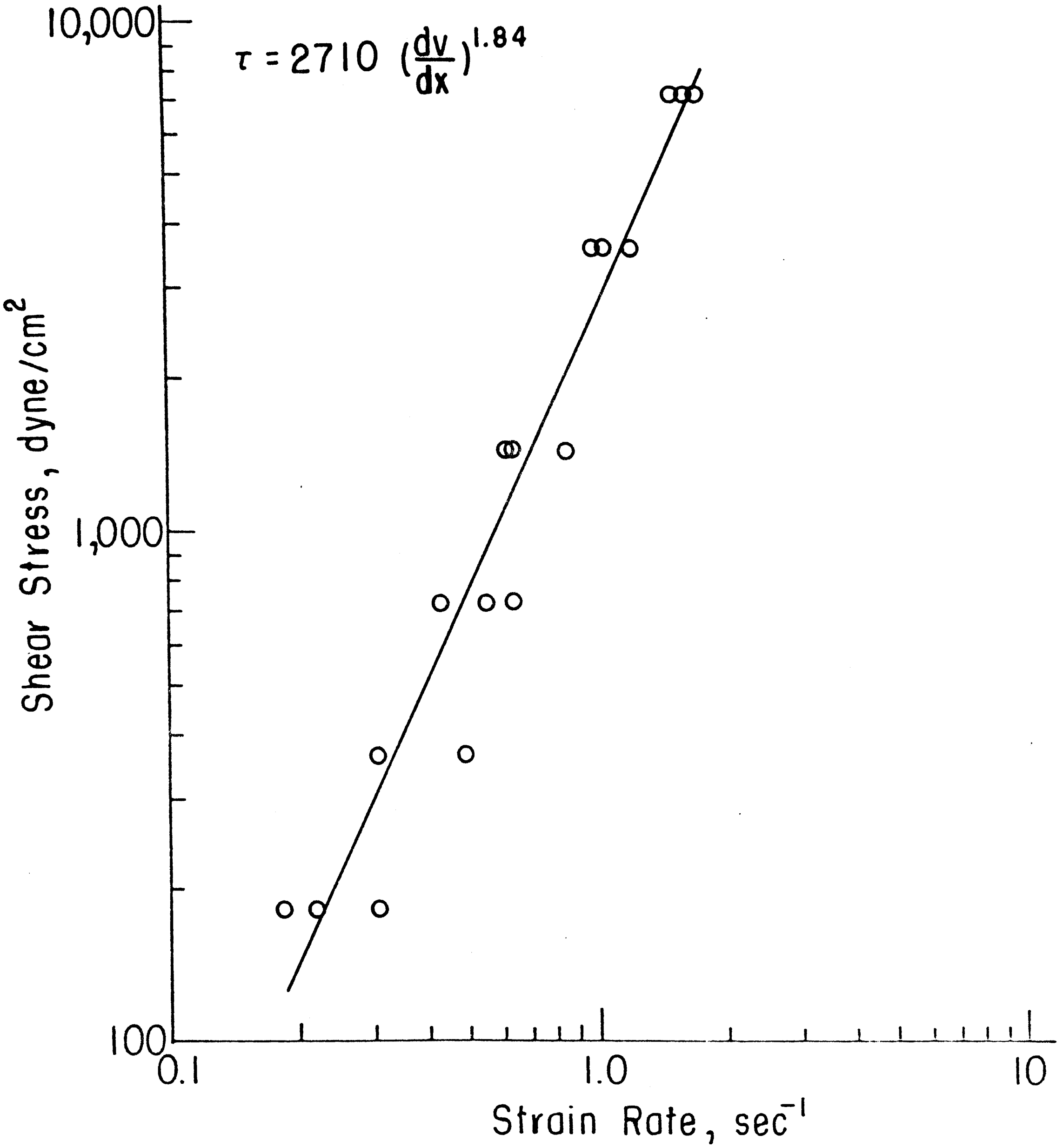

Figure 30. Shear Stress-Strain Rate Relationship for Mass Fraction of 2.233 lit/kg and 4mm Corn Particle Size at 200°F

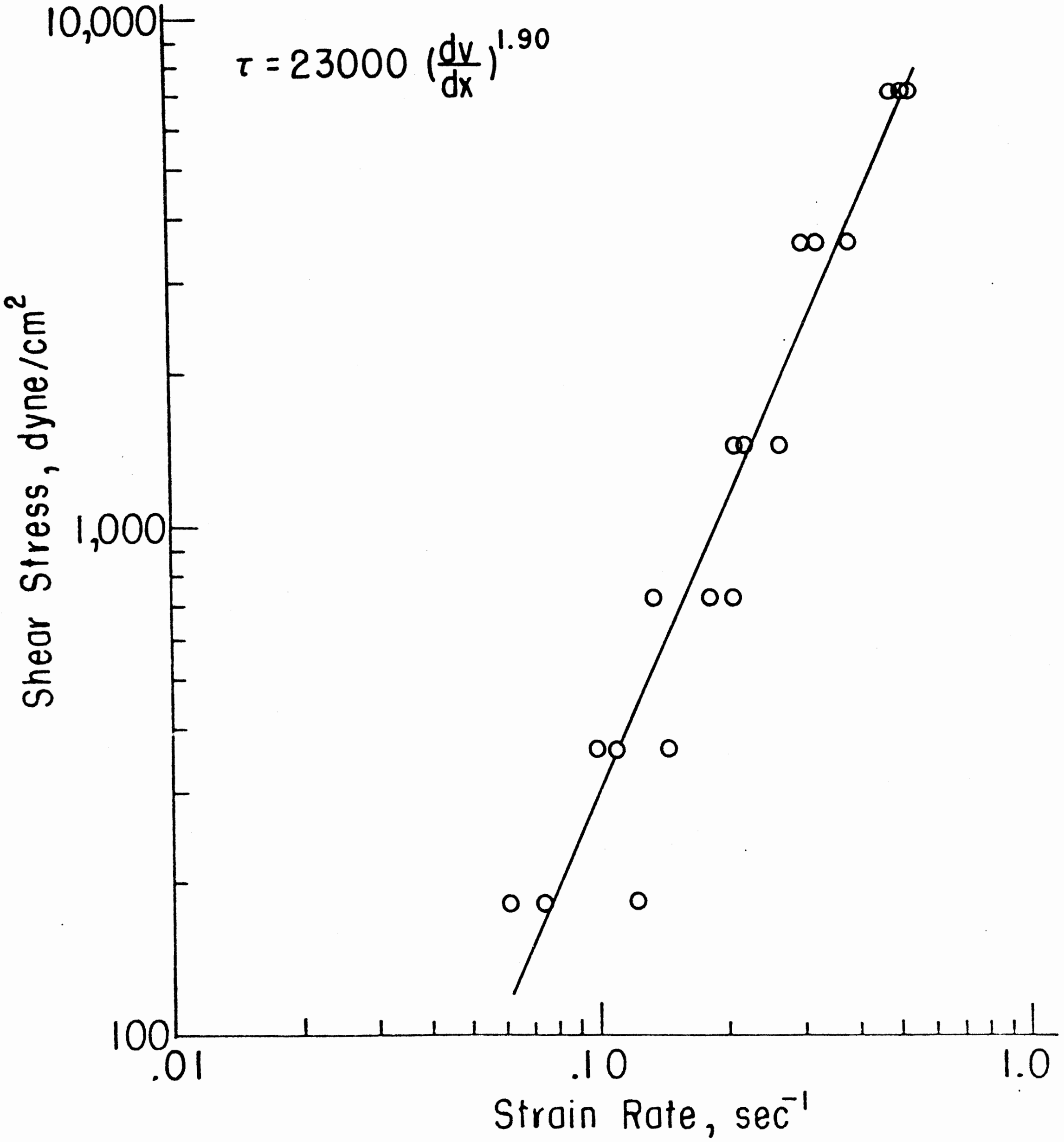

Figure 31. Shear Stress-Strain Rate Relationship for Mass Fraction of 2.680 lit/kg and 4mm Corn Particle Size at 165°F

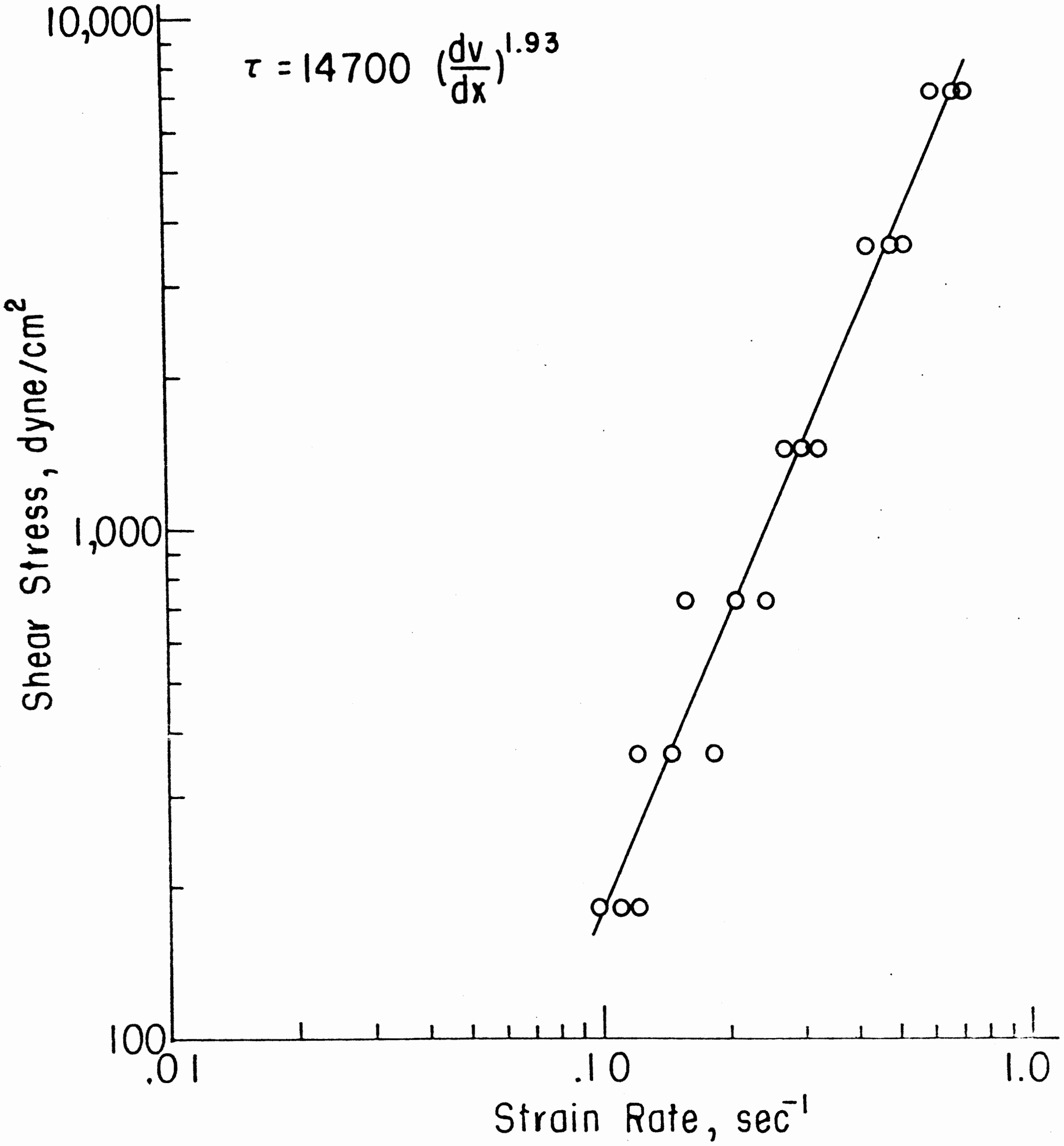

Figure 32. Shear Stress-Strain Rate Relationship for Mass Fraction of 2.680 lit/kg and 4mm Corn Particle Size at 200°F

TABLE X

VALUES OF k AND n IN EQUATION 6 FOR THREE MASS FRACTIONS AT 165 AND 200°F

Mass Fraction lit/kg	Temperature °F	k $\frac{\text{dyne-sec}^n}{\text{cm}^2}$	n dimensionless
1.787	165	1740	1.57
	200	743	1.71
2.233	165	4640	1.81
	200	2710	1.84
2.680	165	23,000	1.90
	200	14,700	1.93

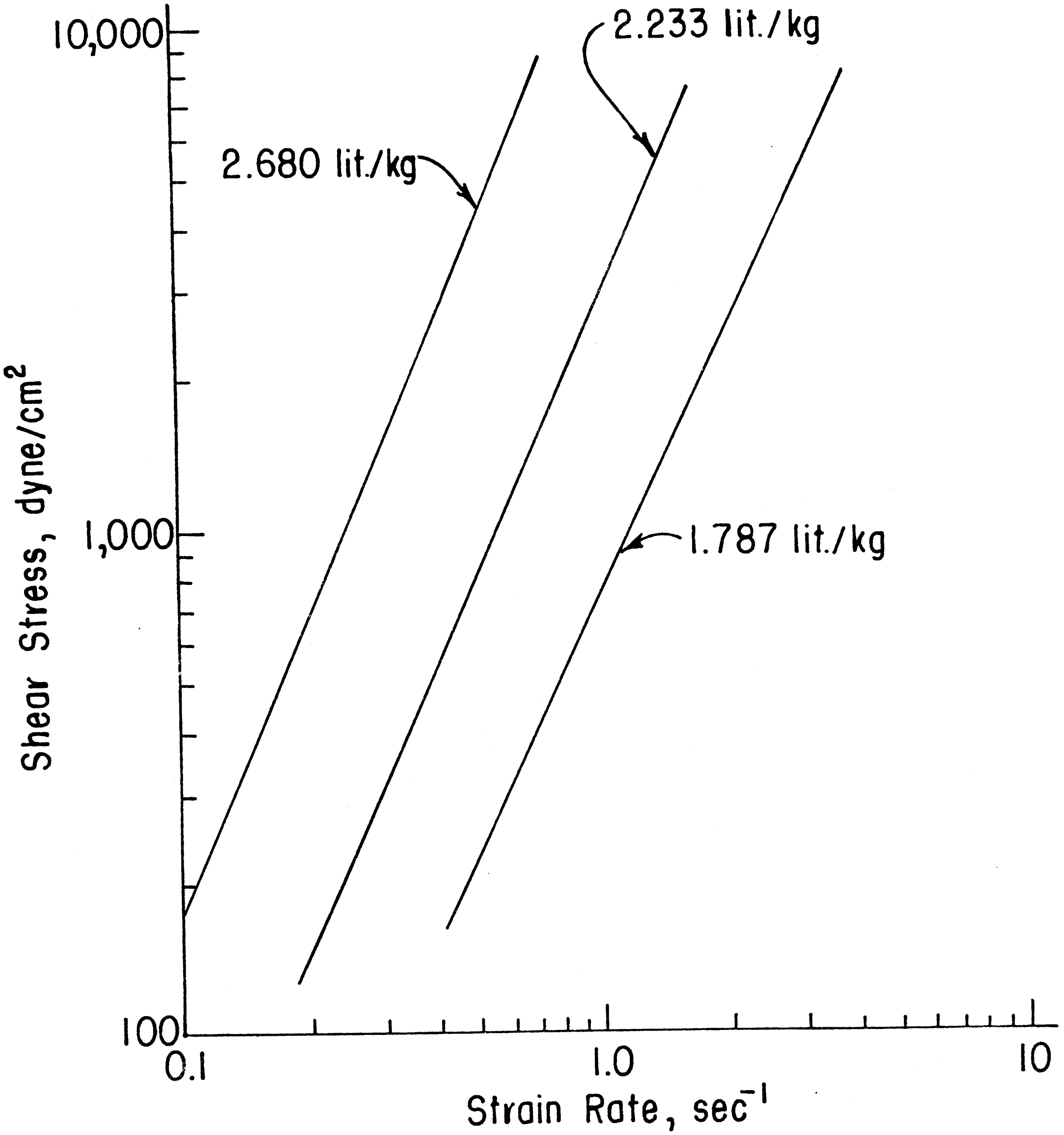

Figure 33. Shear Stress-Strain Rate Relationship for Three Levels of Mass Fraction at 165°F, Using 4mm Ground Corn Mash

plotted in Figure 34 and defined by equation 12:

$$k = a_1 \, (MF)^{b_1} \tag{12}$$

where: k = viscometric index, $\frac{\text{dyne-sec}^n}{\text{cm}^2}$

MF = mass fraction, lit/kg

a_1 = index, $\frac{\text{dyne-sec}^n}{\text{cm}^2} \, (\text{kg/lit})^{b_1}$

b_1 = dimensionless coefficient

Likewise, the slope of the log-log plot of shear stress vs. strain rate curve in Figure 33 is a function of mass fraction of corn to total mass of the slurry. The functional relationship between the exponent, n, and mass fraction for corn slurries at 165°F is plotted in Figure 35 and described by equation 13:

$$n = a_2 \, (MF)^{b_2} \tag{13}$$

n = viscometric coefficient, dimensionless

MF = mass fraction, lit/kg

a_2 = index, $(\text{kg/lit})^{b_2}$

b_2 = dimensionless coefficient

Evaluation of the coefficients a_1 and a_2 and the exponents b_1 and b_2 from Figures 34 and 35 yields equations 14 and 15 for the viscometric coefficient k and viscometric index n for corn-water slurries at 165°F.

$$k = 39.5 \, (MF)^{6.30} \tag{14}$$

$$n = 1.20 (MF)^{0.476} \tag{15}$$

Shear stress-strain rate relationships for three levels

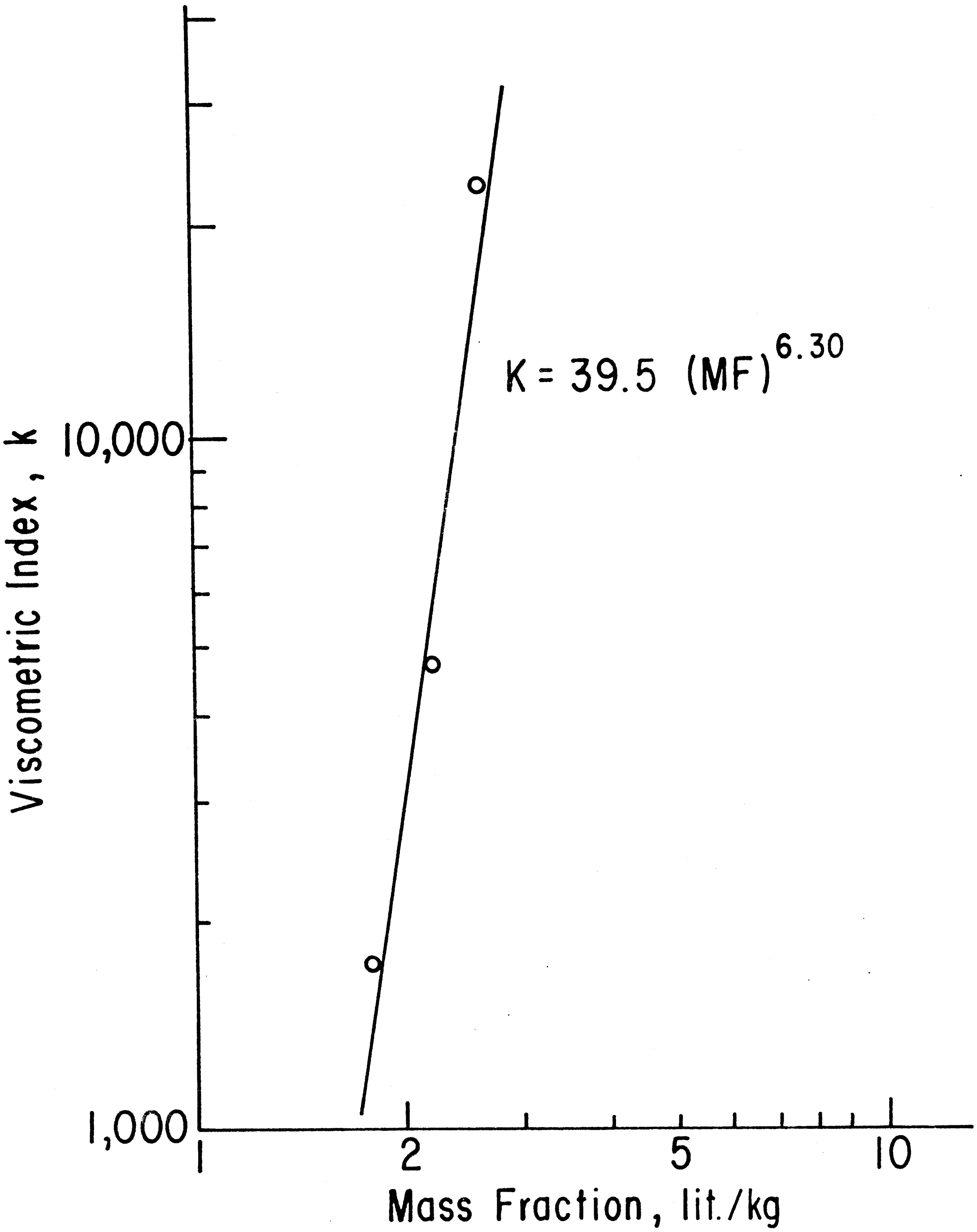

Figure 34. Mass Fraction - Consistency Index (k) Relationship for 4mm Ground Corn at 165°F

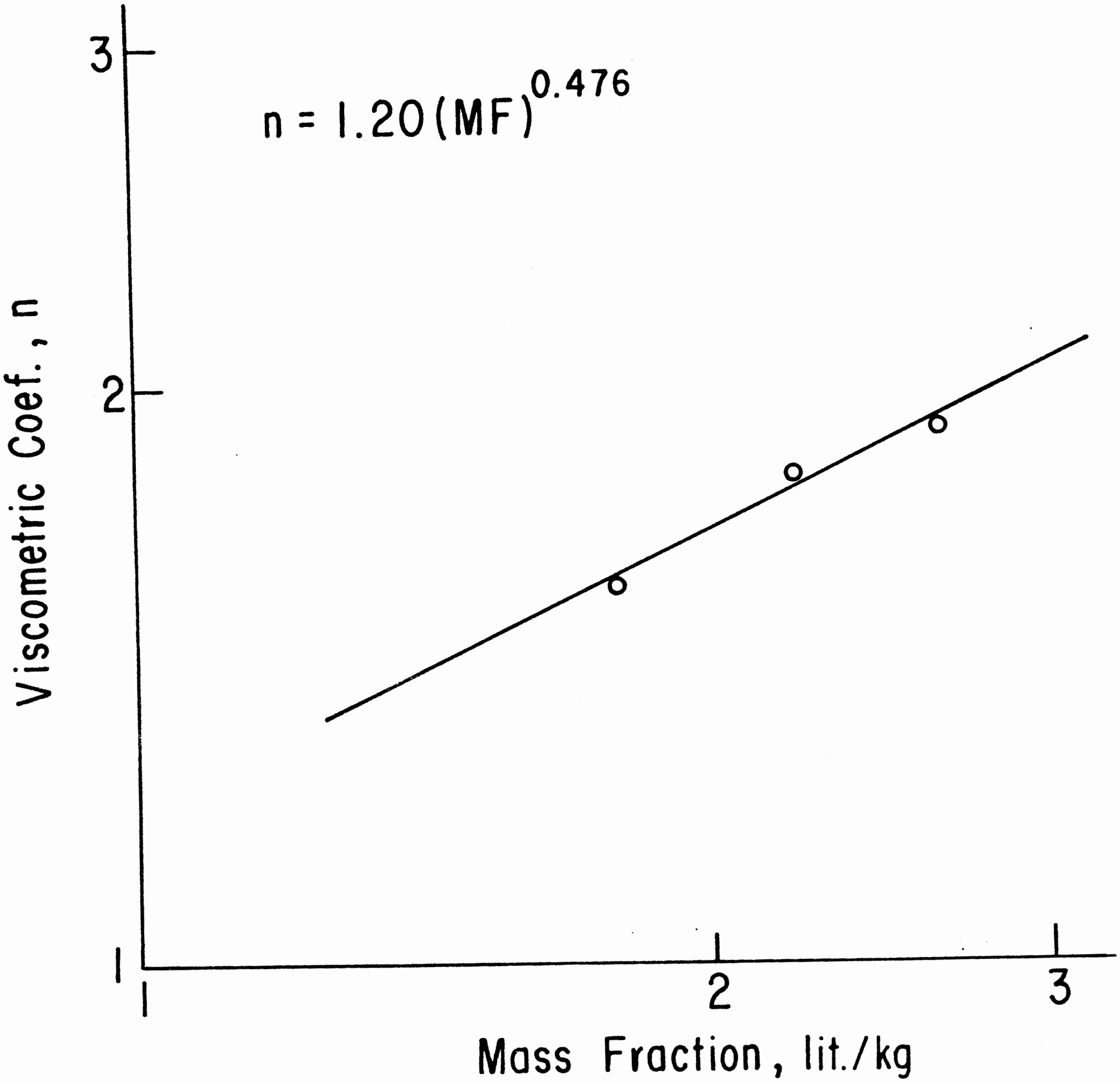

Figure 35. Mass Fraction - Flow Behaviour Index (n) Relationship for 4mm Ground Corn at 165°F

of mass fraction at 200°F are plotted in Figure 36. Effects of mass fraction of corn to total slurry mass on the viscometric properties, k and n, are described by equations 12 and 13 above. Evaluation of the coefficients a_1 and a_2 and exponents b_1 and b_2 from Figure 36 are plotted in Figures 37 and 38 on logarithmic coordinates and described by equations 16 and 17:

$$k = 9.68(MF)^{7.30} \tag{16}$$

$$n = 1.44(MF)^{0.300} \tag{17}$$

Equation 14 and 15 define the viscometric coefficient, k, and viscometric exponent, n, shown in equation 6 for corn- water slurries at 165°F. Likewise, equation 16 and 17 define the viscometric coefficient, k, and viscometric exponent, n, again as defined by equation 6 for corn-water slurries at 200°F.

Combining equations 6, 14 and 15 yields a general shear stress-strain rate relationship for corn water slurries during cooking for the ethanol process at 165°F. The equation is:

$$\tau = [39.5\ (MF)^{6.30}] * \bar{\gamma}^{[1.20(MF)^{0.476}]} \tag{18}$$

Likewise, combining equations 9, 14 and 15 yields the general shear stress-strain rate relationship for corn water slurries at 200°F during cooking for ethanol production. It yields equation 19:

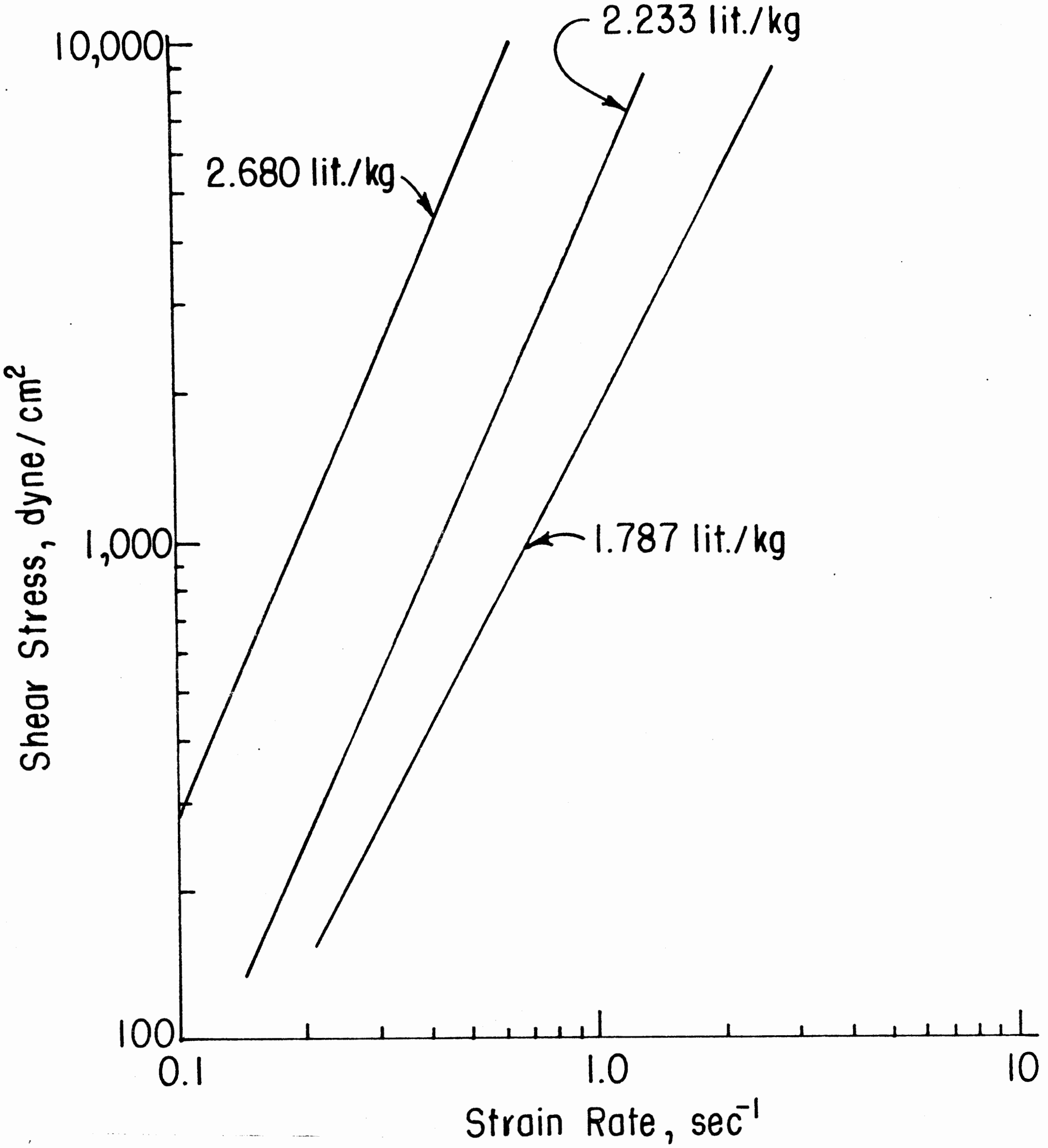

Figure 36. Shear Stress-Strain Rate Relationship for Three Levels of Mass Fraction at 200°F Using 4mm Ground Corn Mash

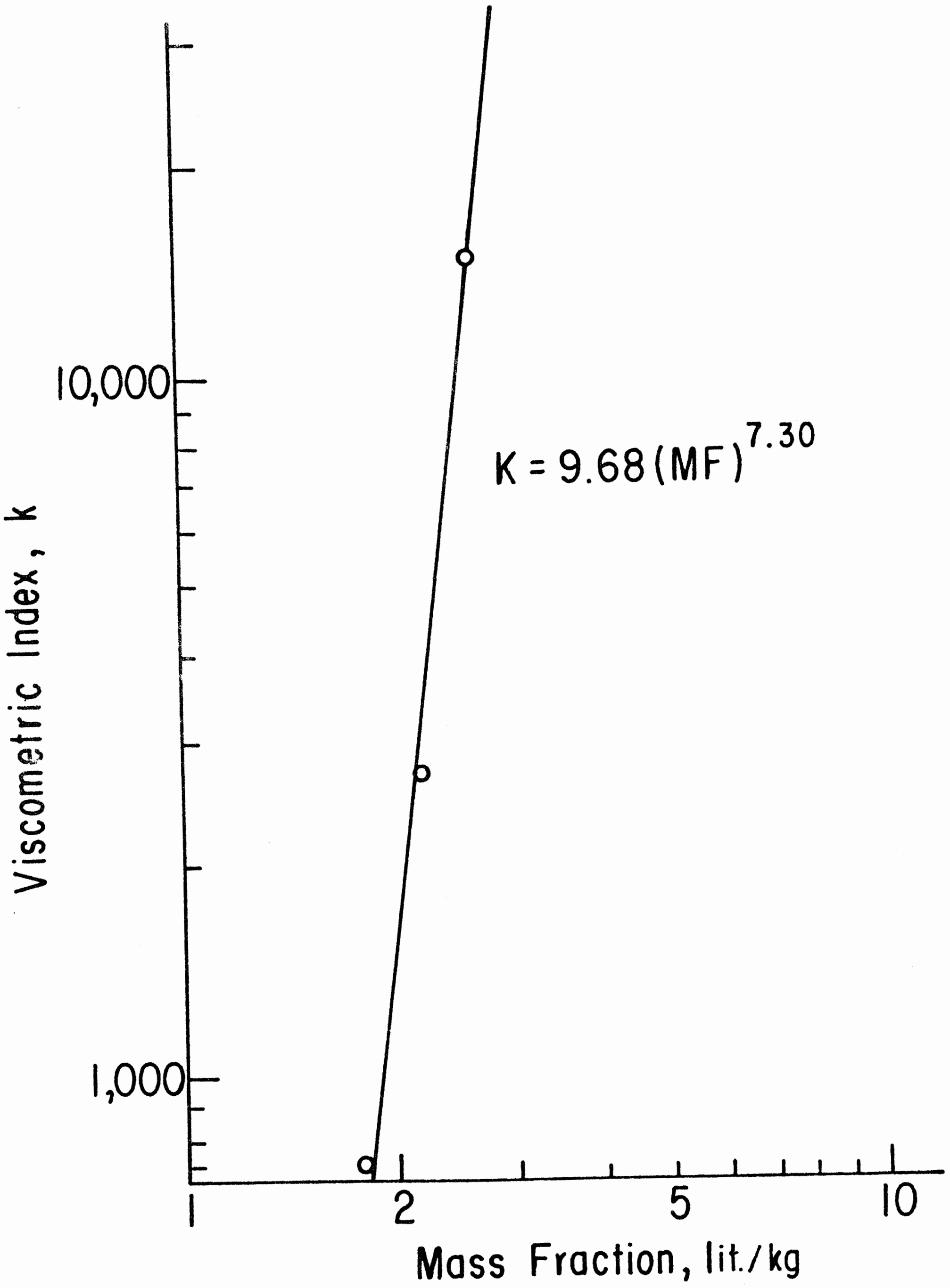

Figure 37. Mass Fraction - Consistency Index (k) Relationship for 4mm Corn Ground at 200°F

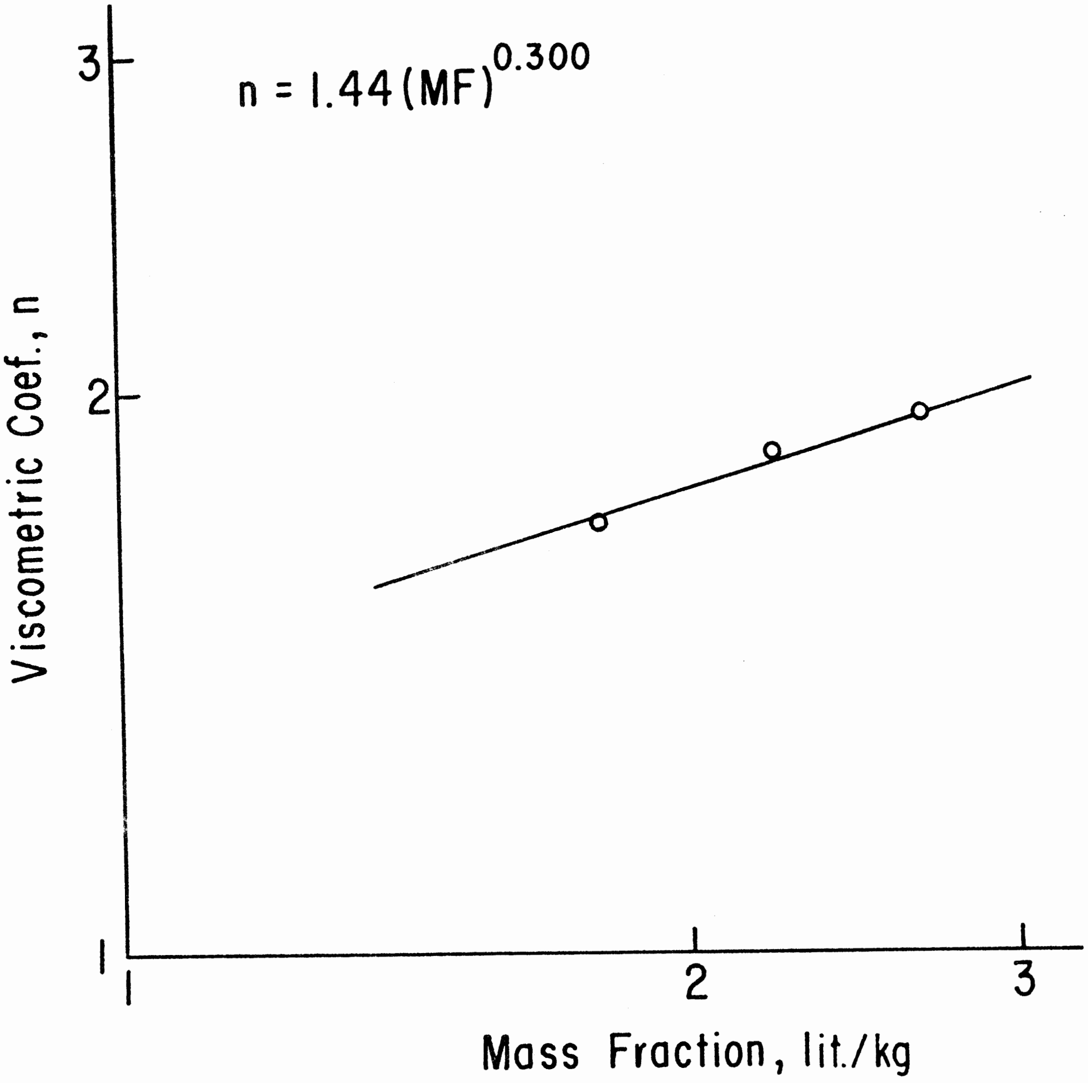

Figure 38. Mass Fraction-Flow Behavior Index (n) Relationship for 4mm Corn Ground at 200°F

$$\tau = [9.68(MF)^{7.30}] * \bar{\gamma}^{[1.44(MF)^{0.300}]} \qquad (19)$$

Therefore, viscometric properties of corn water slurries during cooking are defined for two different temperatures by equations 18 and 19. The 200°F point was selected as previously indicated because this is the maximum temperature reached during batch cooking of corn water slurries while producing fuel grade ethanol at atmospheric pressure. Likewise, equation 18 defines maximum viscometric properties experienced during cooking of corn water slurries. Utilization of equation 18 and 19 allows determination of peak maximum viscometric properties of non-newtonian corn water slurry during cooking and determination of viscometric properties for the same slurry at maximum cooking temperatures of 200°F. These properties can be utilized in determining agitation and pumping power requirements during processing of corn water slurries for ethanol production.

CHAPTER V

SUMMARY AND CONCLUSION

Viscometric properties of corn-water slurries during cooking for ethanol production were evaluated as a function of mass fraction and ground corn particle size. The effect of starch granule swelling and disintegration during cooking on apparent viscosity of slurries were studied. Maximum apparent viscosity was observed to occur at 165°F-170°F. Maximum cooking temperature was 200°F, since it is typical of maximum cooking temperature in batch processes at one atmosphere.

Analysis of variance and Duncan's Multiple Range Test were performed on peak apparent viscosity and apparent viscosity at 200°F. Shear Stress-Strain Rate relationships at these temperatures (165°F and 200°F) were measured using Brookfield's rotational viscometer. The LV#2 cylindrical spindle was used and dial displacements were recorded at various angular velocities of the spindle. Shear Stress and Strain Rate values were calculated from these data using equation 9 and 11, and viscometric parameters were evaluated.

Conclusion

Interpretation of the experimental data resulted in the following conclusions:

1) Mass fraction is the most important parameter affecting apparent viscosity of the corn-water slurry during cooking for ethanol production.
2) Particle size was found to have no significant effect on viscometric properties of corn-water slurries during cooking for ethanol production.
3) The corn-water slurries were found to be power law non-newtionian fluids at temperatures of 165°F and 200°F and are described by equation 6:

$$\tau = K\left(\frac{dv}{dx}\right)^n \qquad (6)$$

where τ = Shear Stress, dyne/cm^2

$\frac{dv}{dx}$ = Strain rate, sec^{-1}

K = viscometric index, $\frac{\text{dyne-sec}^n}{\text{Cm}^2}$

n = viscometric coefficient, dimensionless

since the slope of the line, n, is greater than unity, the corn-water slurry is classified as dilatant fluid.

4) The viscometric index, k, is a function of corn to total slurry mass. The effect of mass fraction on the viscometric index, k, is plotted in logarithmic coordinates and defined by equation 14 and 16:

$$K = 39.5\ (MF)^{6.30},\ \text{at } 165°F \qquad (14)$$

and $$K = 9.68\ (MF)^{7.30},\ \text{at } 200°F \qquad (16)$$

where K = viscometric index, $\frac{\text{dyne-sec}^n}{\text{Cm}^2}$

MF = mass fraction, lit/kg

5) The viscometric coefficient, n, is a function of mass fraction of corn to the total mass of slurry. The functional relationship between the exponent, n, and mass fraction plots as a straight line in logarithmic coordinates and is described by:

$$n = 1.20\ (MF)^{0.476},\ \text{at } 165°F \qquad (15)$$

$$n = 1.44\ (MF)^{0.300},\ \text{at } 200°F \qquad (17)$$

where n = viscometric coefficient, dimensionless

MF = mass fraction, lit/kg

6) An equation for shear stress-strain rate relationship for corn water slurries during cooking for ethanol production, using 4 mm ground corn at 165°F was obtained. The equation describing this relationship at 165°F is:

$$\tau = [39.5(MF)^{6.30}] * \bar{\gamma}^{[1.20(MF)^{0.476}]} \qquad (18)$$

Likewise at 200°F the equation was found to be:

$$\tau = [9.68(MF)^{7.30}] * \bar{\gamma}^{[1.44(MF)^{0.300}]} \qquad (19)$$

Suggestions for Further Study

Viscometric properties of other types of cereal grain slurries such as wheat and milo should be studied.

Further testing with a wider range of particle size and mass fraction of corn to total slurry is necessary to test the reliability of equations developed for viscometric index, k (equation 12) and viscometric coefficient, n (equation 13).

Further study should be performed on reliability of equations 18 and 19 for designing the agitation system of corn water slurry during cooking for ethanol production.

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APPENDIX A

MASS DISTRIBUTION OF 3, 4 AND 5 MM GROUND CORN FOR THREE REPLICATIONS AFTER 10 MINUTES OF SHAKING

TABLE XI

PARTICLE SIZE DISTRIBUTION OF 3 MM GROUND CORN AFTER 10 MINUTES OF SHAKING

Tyler Sieve #	Mass of Corn, Grams 1st Replication	2nd Replication	3rd Replication	Average
14	36.68	36.08	40.66	37.81
28	34.85	34.59	34.39	34.61
40	7.35	7.47	6.39	7.07
48	9.36	8.45	6.95	8.25
60	1.42	1.35	2.14	1.64
100	6.25	6.95	5.58	6.26
PAN	0.22	0.18	0.04	0.15
Total	96.13	95.07	96.15	95.78

TABLE XII

PARTICLE SIZE DISTRIBUTION OF 4 MM GROUND CORN AFTER 10 MINUTES OF SHAKING

	Mass of Corn, Grams			
Tyler Sieve #	1st Replication	2nd Replication	3rd Replication	Average
14	39.88	51.09	49.44	46.80
28	28.89	27.78	29.16	28.61
40	7.62	5.52	6.66	6.60
48	13.28	5.64	7.14	8.69
60	0.96	1.73	1.16	1.28
100	6.54	5.79	6.38	6.24
PAN	0.75	0.59	1.00	0.78
Total	97.92	98.14	100.94	99.00

TABLE XIII

PARTICLE SIZE DISTRIBUTION OF 5 MM GROUND CORN AFTER 10 MINUTES OF SHAKING

	Mass of Corn, Grams			
Tyler Sieve #	1st Replication	2nd Replication	3rd Replication	Average
8	22.3	21.13	19.9	21.11
14	51.1	49.53	50.07	50.23
28	16.27	17.13	17.18	16.86
48	6.12	7.07	6.92	6.70
60	2.48	2.17	2.27	2.31
100	3.12	3.99	3.99	3.70
PAN	1.04	1.07	0.88	1.00
Total	102.43	102.09	101.21	101.91

APPENDIX B

APPARENT VISCOSITY OF GROUND CORN MASH AT 150-200°F MEASUREMENTS PERFORMED USING RV#3 SPINDLE AT 10 RPM

TABLE XIV

APPARENT VISCOSITY OF 3 MM GROUND CORN MASH, USING 12 GALLONS OF WATER PER BUSHEL OF CORN (1.787 lit/kg) AT 150-200°F

Sample	Avg. Time min:sec	Temperature °F	App. Viscosity, Centipoise. Replications: 1st	2nd	3rd
1	6:42	150	170	150	170
2	8:35	155	310	740	310
3	10:37	160	1300	3400	3200
4	13:07	165	3100	9100	7600
5	16:13	170	6300	5700	5900
6	18:48	175	5600	4100	4600
7	21:13	180	5300	3500	4400
8	24:13	185	4500	3700	4800
9	27:22	190	4300	4000	5300
10	30:08	195	4400	3200	4900
11	34:35	200	6400	3800	6700

TABLE XV

APPARENT VISCOSITY OF 3 MM GROUND CORN MASH, USING 15 GALLONS OF WATER PER BUSHEL OF CORN (2.233 lit/kg) AT 150-200°F

Sample	Avg. Time min:sec	Temperature °F	App. Viscosity, Centipoise Replications: 1st	2nd	3rd
1	4:22	150	14	8	9
2	6:52	155	90	66	100
3	10:08	160	450	720	290
4	13:03	165	800	980	490
5	16:02	170	860	1100	1000
6	20:02	175	720	1000	1000
7	24:27	180	740	780	850
8	28:43	185	850	850	900
9	35:58	190	780	880	1100
10	41:22	195	720	900	960
11	49:27	200	800	1060	860

TABLE XVI

APPARENT VISCOSITY OF 3 MM GROUND CORN MASH, USING 18 GALLONS OF WATER PER BUSHEL OF CORN (2.680 lit/kg) AT 150-200°F

Sample	Avg. Time min:sec	Temperature °F	App. Viscosity, Centipoise Replications: 1st	2nd	3rd
1	6:22	150	6	6	6
2	8:25	155	15	11	11
3	10:42	160	200	270	370
4	12:58	165	1200	640	700
5	14:47	170	960	820	720
6	16:58	175	800	680	700
7	19:25	180	820	630	780
8	21:47	185	840	740	900
9	25:02	190	1000	820	1000
10	27:40	195	1100	840	980
11	33:06	200	1200	1400	1800

TABLE XVII

APPARENT VISCOSITY OF 4 MM GROUND CORN MASH, USING 12 GALLONS OF WATER PER BUSHEL OF CORN (1.787 lit/kg) AT 150-200°F

Sample	Avg. Time min:sec	Temperature °F	App. Viscosity, Centipoise Replications: 1st	2nd	3rd
1	6:28	150	13	150	130
2	8:00	155	230	830	400
3	10:07	160	1200	3000	3200
4	13:18	165	2700	8200	6400
5	16:35	170	2000	3200	8200
6	19:07	175	2100	2900	3200
7	22:28	180	2200	2400	2400
8	25:48	185	2200	2200	2300
9	28:40	190	1700	2700	2800
10	32:13	195	2100	2700	3200
11	38:25	200	6700	3800	3400

TABLE XVIII

APPARENT VISCOSITY OF 4 MM GROUND CORN MASH, USING 15 GALLONS OF WATER PER BUSHEL OF CORN (2.233 lit/kg) AT 150-200°F

Sample	Avg. Time min:sec	Temperature °F	App. Viscosity, Centipoise Replications: 1st	2nd	3rd
1	6:05	150	15	8	9
2	8:18	155	120	28	97
3	10:53	160	960	400	760
4	13:27	165	2300	5000	1600
5	16:28	170	3000	2600	1600
6	19:47	175	2300	1500	1700
7	22:18	180	1800	1800	1300
8	24:28	185	1800	1500	1500
9	27:02	190	2000	1200	1300
10	30:12	195	2300	1500	1200
11	34:42	200	2600	1800	1700

TABLE XIX

APPARENT VISCOSITY OF 4 MM GROUND CORN MASH, USING 18 GALLONS OF WATER PER BUSHEL OF CORN (2.680 lit/kg) AT 150-200°F

Sample	Avg. Time min:sec	Temperature °F	App. Viscosity, Centipoise Replications: 1st	2nd	3rd
1	7:17	150	5	7	6
2	9:02	155	10	11	13
3	11:28	160	800	200	240
4	13:55	165	960	820	440
5	16:10	170	1000	840	770
6	19:17	175	820	790	820
7	22:18	180	700	590	730
8	25:33	185	860	720	760
9	28:53	190	880	770	660
10	32:10	195	1100	740	780
11	37:10	200	1500	860	820

TABLE XX

APPARENT VISCOSITY OF 5 MM GROUND CORN MASH, USING 12 GALLONS OF WATER PER BUSHEL OF CORN (1.787 lit/kg) AT 150-200°F

Sample	Avg. Time min:sec	Temperature °F	App. Viscosity, Centipoise Replications: 1st	2nd	3rd
1	7:53	150	12	84	22
2	10:20	155	120	800	290
3	11:50	160	980	1900	1800
4	14:18	165	1700	4400	3100
5	16:32	170	2000	3900	2800
6	18:43	175	1700	3800	3000
7	21:22	180	1600	3400	2900
8	25:30	185	1300	3700	2300
9	29:40	190	1300	3700	2400
10	34:53	195	2000	3900	3600
11	41:40	200	2700	6800	3800

TABLE XXI

APPARENT VISCOSITY OF 5 MM GROUND CORN MASH, USING 15 GALLONS OF WATER PER BUSHEL OF CORN (2.233 lit/kg) AT 150-200°F

Sample	Avg. Time min:sec	Temperature °F	App. Viscosity, Centipoise Replications: 1st	2nd	3rd
1	7:43	150	9	7	10
2	9:43	155	84	53	250
3	11:50	160	1000	570	540
4	14:00	165	2300	2600	1700
5	16:43	170	2200	1400	2000
6	19:23	175	1800	1100	2000
7	21:48	180	1800	1200	1600
8	25:00	185	1800	1000	1700
9	29:45	190	1600	1800	1300
10	32:57	195	2200	1000	1700
11	38:47	200	2600	2000	1800

TABLE XXII

APPARENT VISCOSITY OF 5 MM GROUND CORN MASH, USING 18 GALLONS OF WATER PER BUSHEL OF CORN (2.680 lit/kg) AT 150-200°F

			App. Viscosity, Centipoise Replications:		
Sample	Avg. Time min:sec	Temperature °F	1st	2nd	3rd
1	7:02	150	5	6	4
2	9:12	155	5	9	8
3	11:23	160	380	150	210
4	14:08	165	540	390	950
5	16:32	170	640	900	820
6	20:05	175	720	760	920
7	23:38	180	840	880	800
8	27:57	185	1000	920	940
9	30:47	190	920	780	900
10	34:58	195	1100	1200	1000
11	43:12	200	1700	1300	1400

APPENDIX C

PLOT OF APPARENT VISCOSITY OF GROUND CORN MASH AT 150-200°F

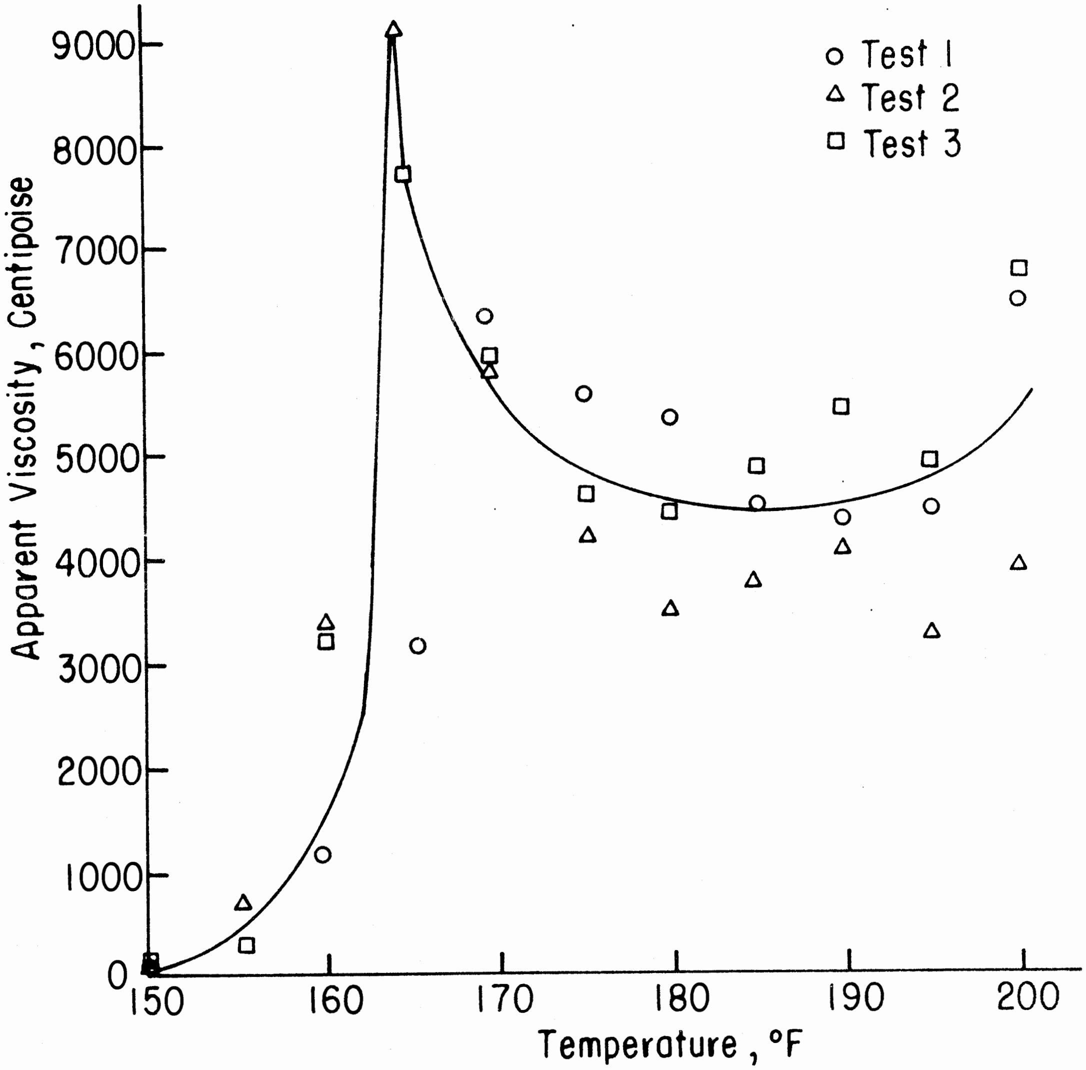

Figure 39. Plot of Apparent Viscosity of 3 mm Ground Corn Mash During Cooking at One Atmosphere. The Solid Mass Fraction is 1.787 lit/kg

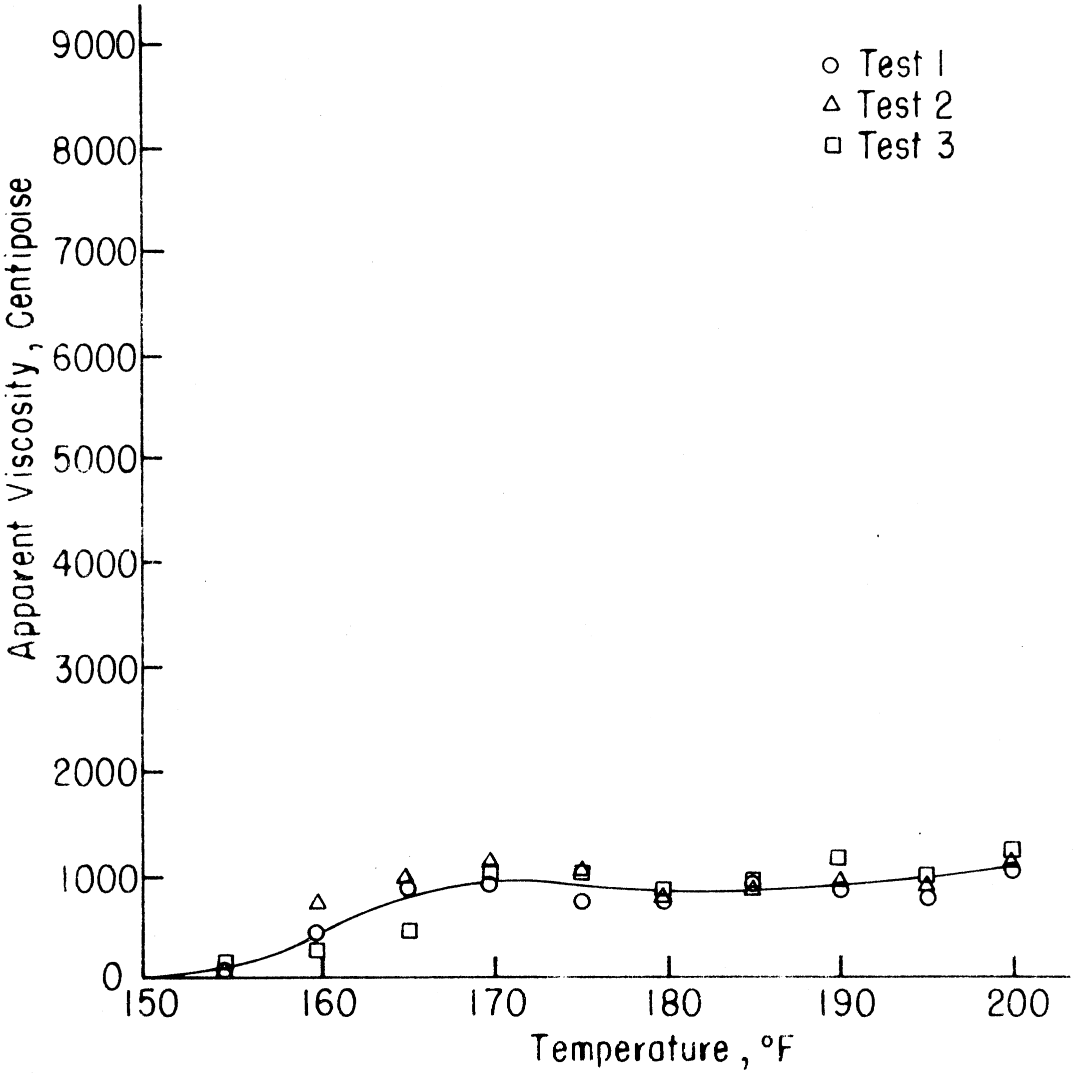

Figure 40. Plot of Apparent Viscosity of 3 mm Ground Corn Mash During Cooking at One Atomsphere. The Solid Mass Fraction is 2.233 lit/kg

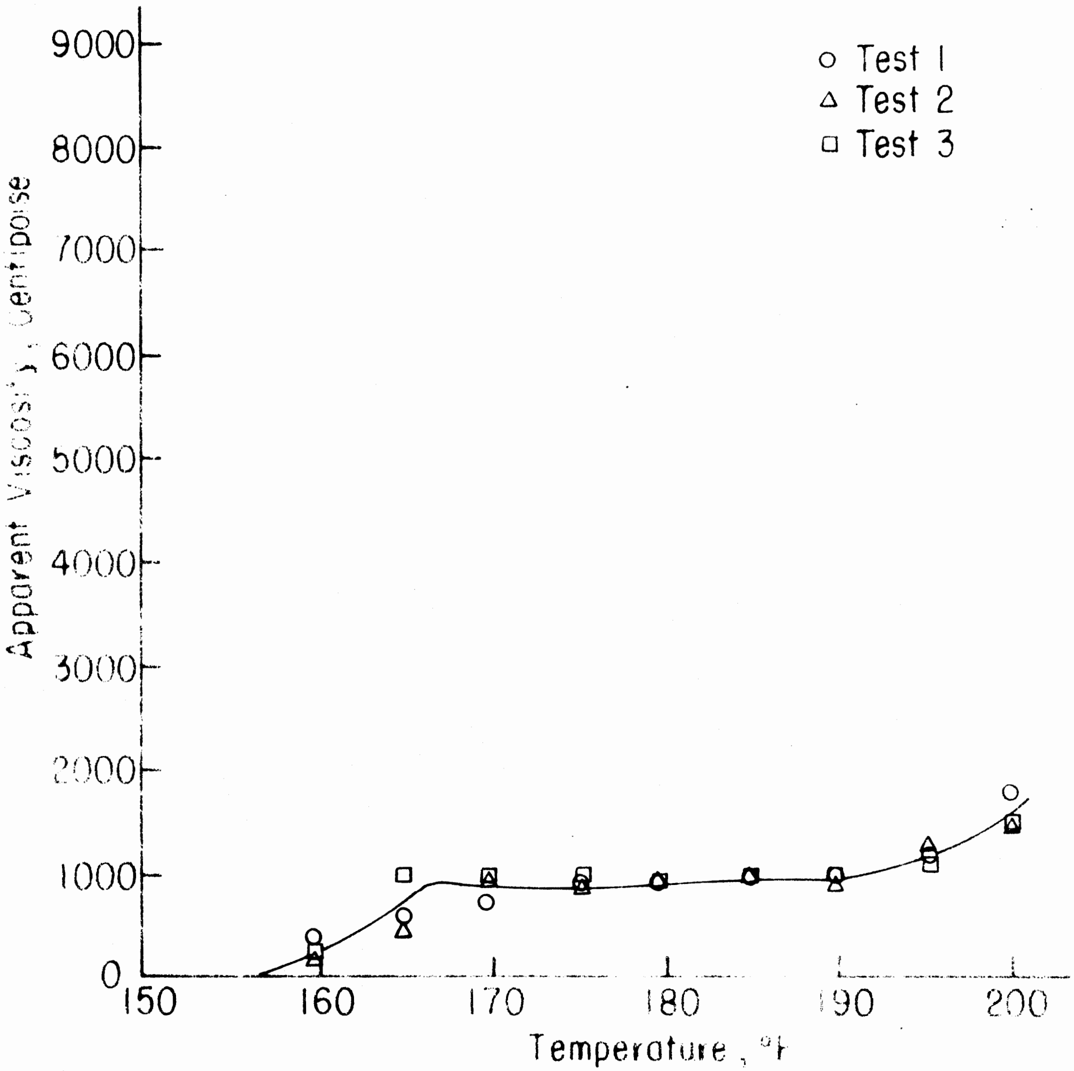

Figure 41. Plot of Apparent Viscosity of 3 mm Ground Corn Mash During Cooking at One Atmo- Atmosphere. The Solid Mass Fraction is 2.680 lit/kg

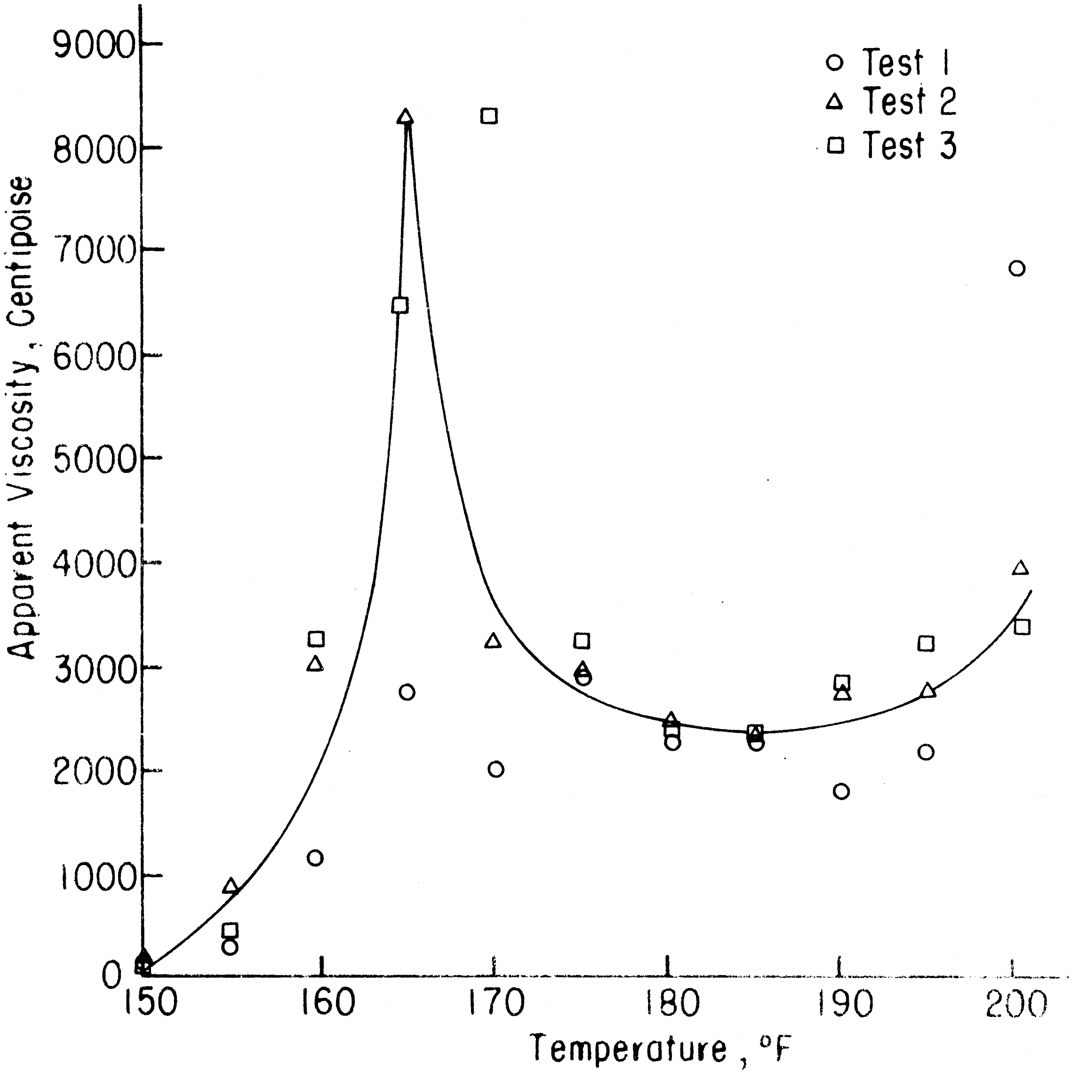

Figure 42. Plot of Apparent Viscosity of 4 mm Ground Corn Mash During Cooking at One Atmosphere. The Solid Mass Fraction is 1.787 lit/kg

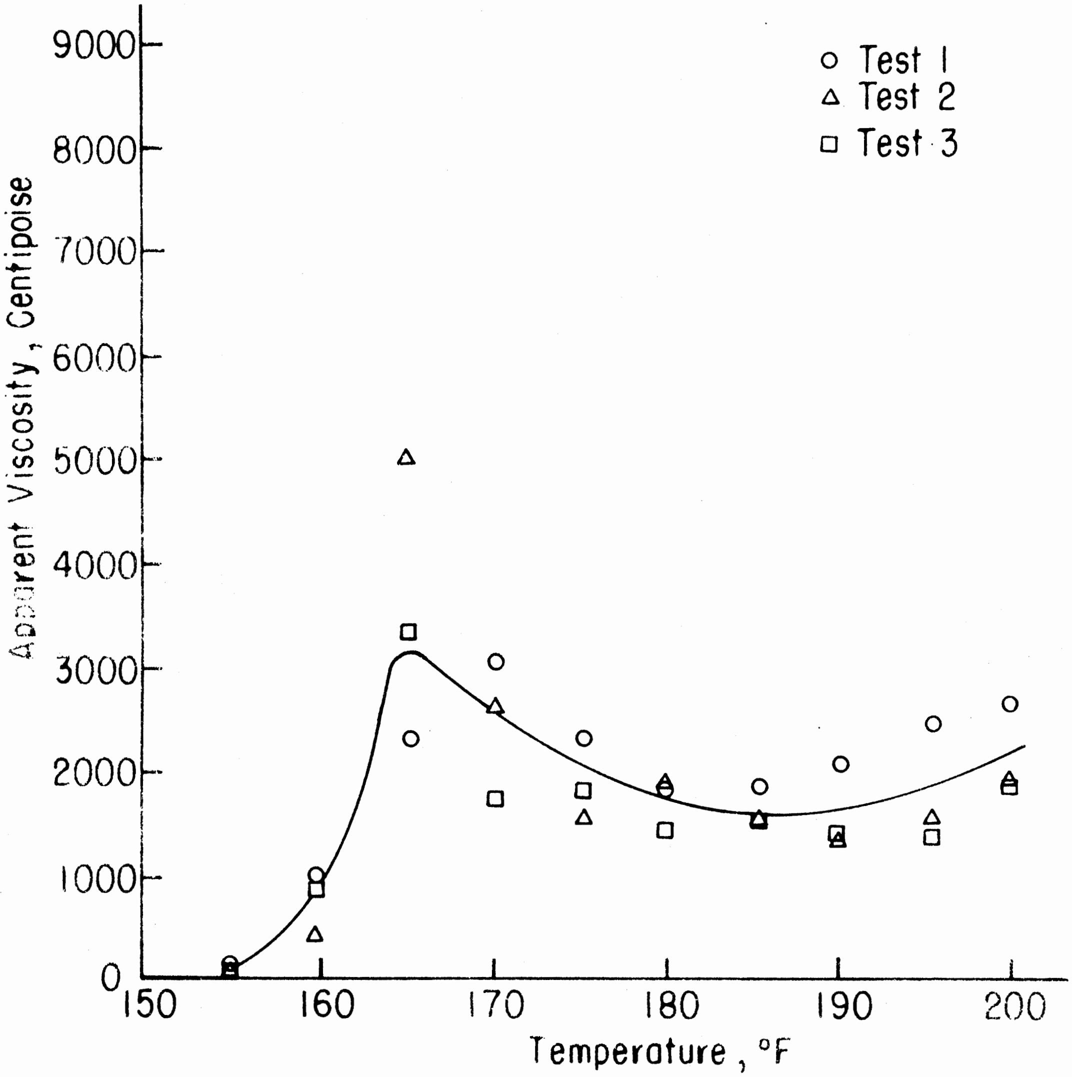

Figure 43. Plot of Apparent Viscosity of 4 mm Ground Corn Mash During Cooking at One Atmosphere. The Solid Mass Fraction is 2.233 lit/kg

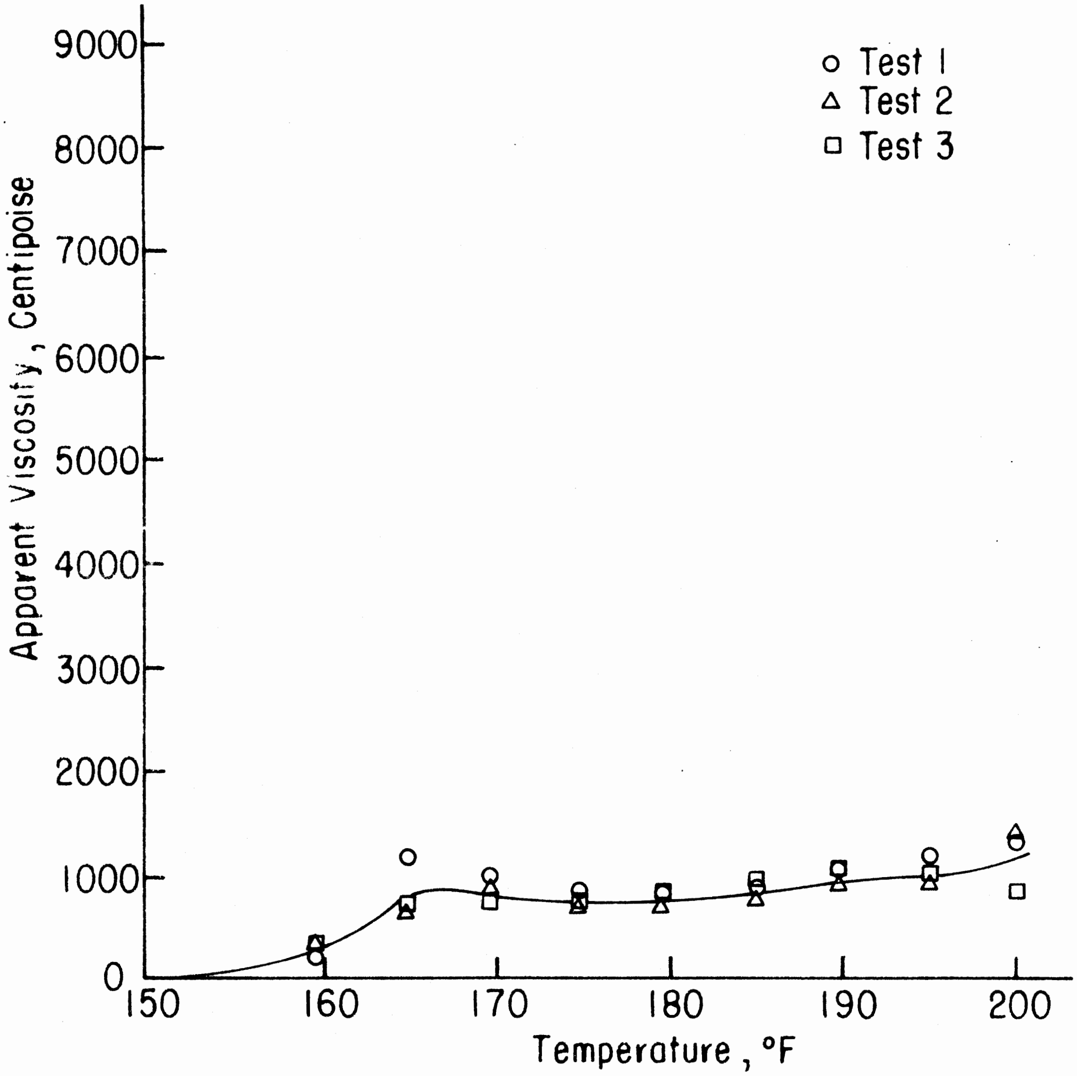

Figure 44. Plot of Apparent Viscosity of 4 mm Ground Corn Mash During Cooking at One Atmosphere. The Solid Mass Fraction is 2.680 lit/kg

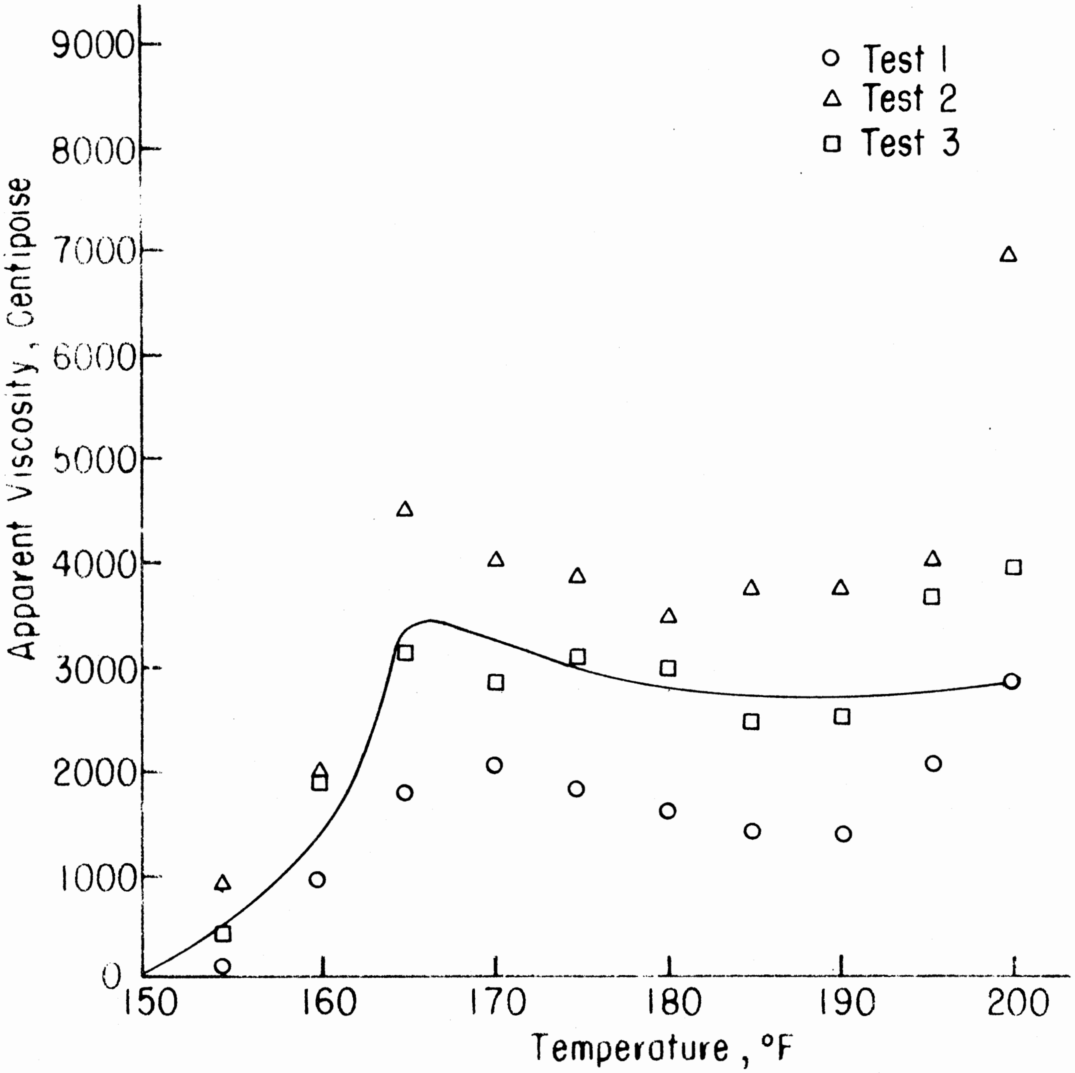

Figure 45. Plot of Apparent Viscosity of 5 mm Ground Corn Mash During Cooking at One Atmosphere. The Solid Mass Fraction is 2.233 lit/kg

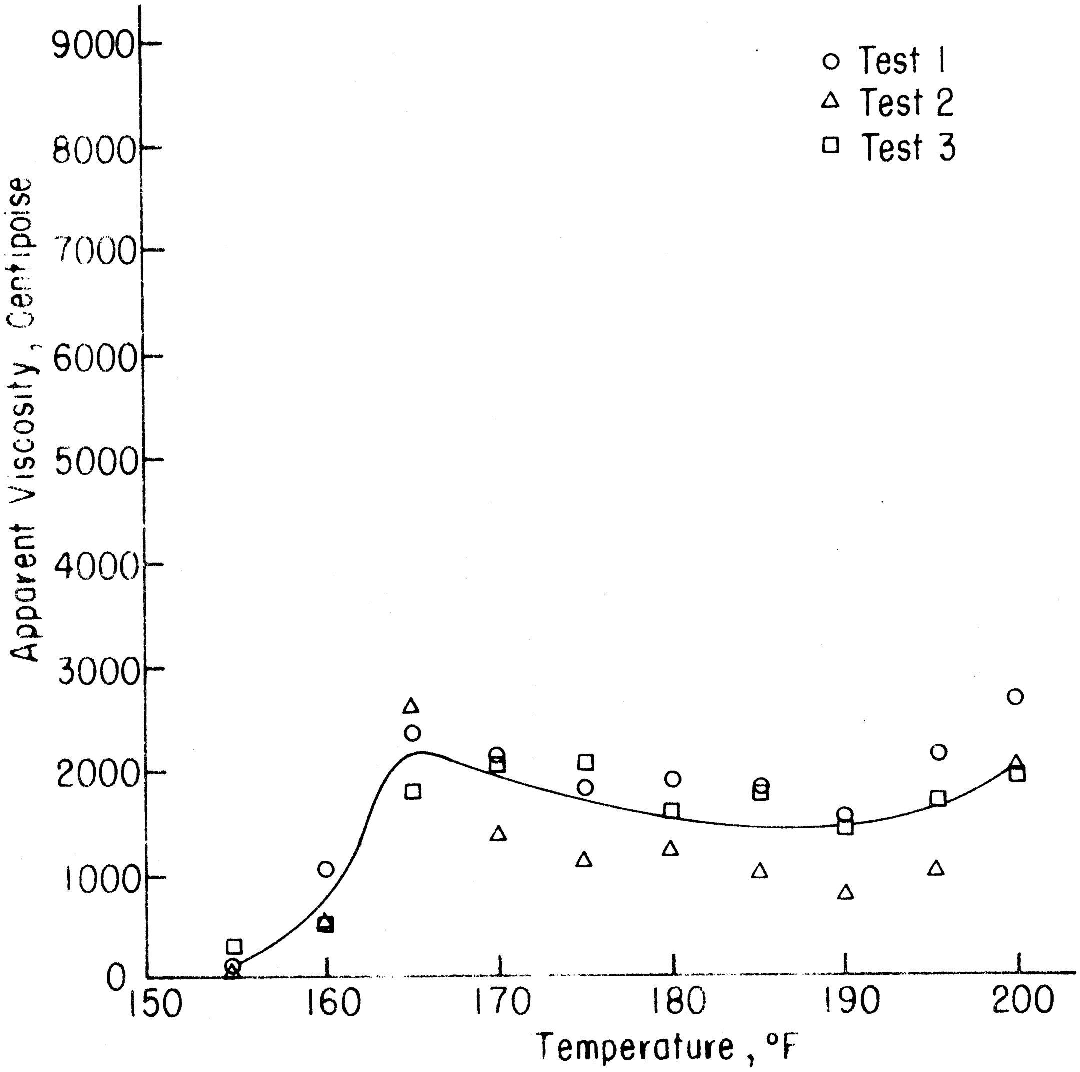

Figure 46. Plot of Apparent Viscosity of 5 mm Ground Corn Mash During Cooking at One Atmosphere. The Solid Mass Fraction is 2.680 lit/kg

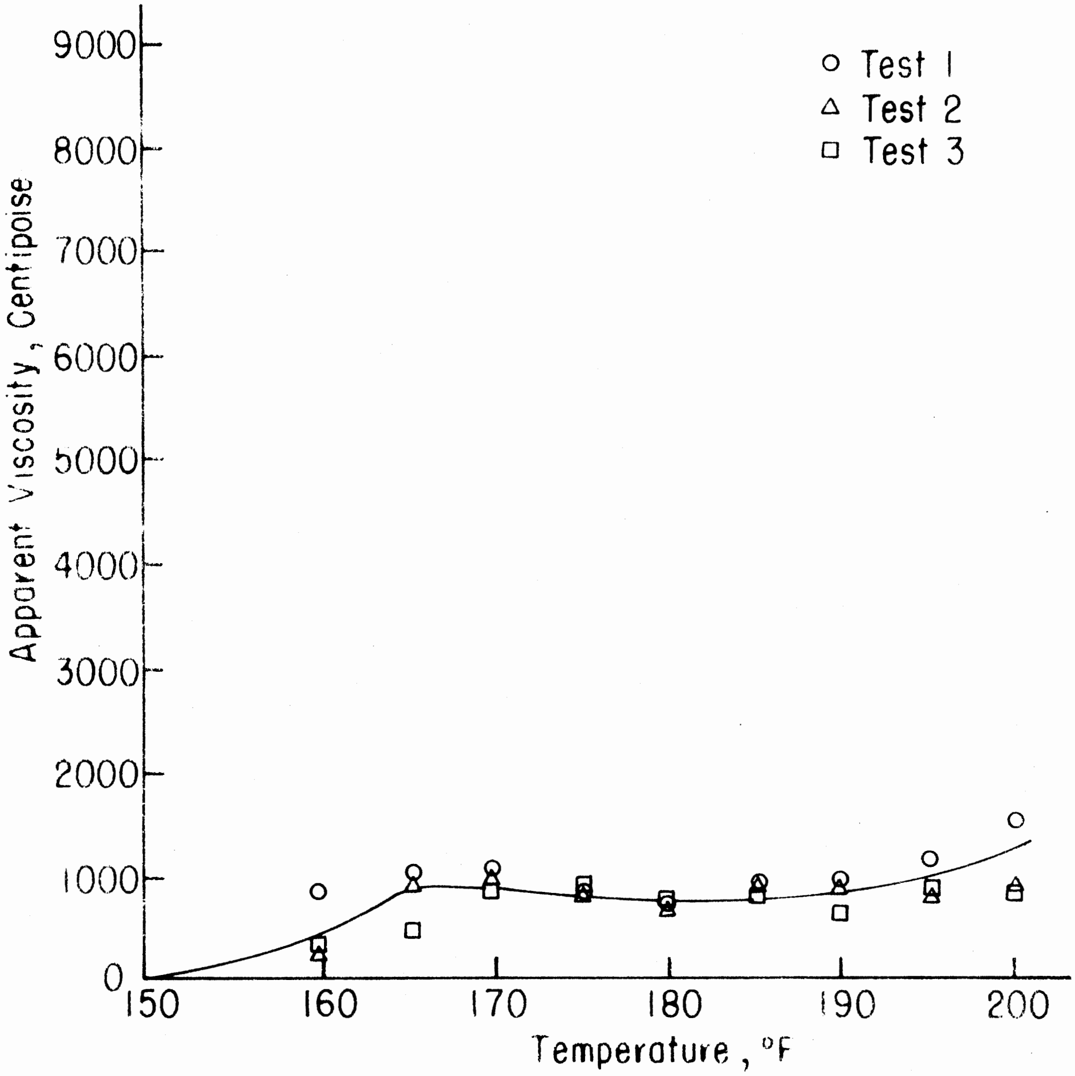

Figure 47. Plot of Apparent Viscosity of 5mm Ground Corn Mash During Cooking at One Atmosphere. The Solid Mass Fraction is 2.680 lit/kg

APPENDIX D

SHEAR STRESS AND STRAIN RATE VALUES CALCULATED BY EQUATIONS 9 AND 11 RESPECTIVELY. RPM AND DIAL READING ARE REPORTED. 4 MM GROUND CORN WAS USED

TABLE XXIII

SHEAR STRESS AND STRAIN RATE VALUES FOR 1.787 lit/kg AT 165°F; USING 4 MM [illegible]

RPM	Shear Stress dyne/Cm^2	Dial Readings 1st repl	Dial Readings 2nd repl	Dial Readings 3rd repl	Strain Rate, Sec^{-1} 1st repl	Strain Rate, Sec^{-1} 2nd repl	Strain Rate, Sec^{-1} 3rd repl
2.5	186	2.0	2.0	1.7	0.248	0.248	0.211
5	372	3.3	3.2	3.0	0.409	0.397	0.372
10	744	5.0	4.7	4.5	0.62	0.583	0.558
20	1490	7.0	7.0	7.0	0.868	0.868	0.868
50	3720	12.0	12.0	13.5	1.49	1.49	1.67
100	7440	19.3	20.0	23.5	2.39	2.48	2.92

TABLE XXIV

SHEAR STRESS AND STRAIN RATE VALUES FOR 1.787 lit/kg AT 200°F; USING 4 MM GROUND CORN

RPM	Shear Stress dyne/Cm^2	Dial Readings			Strain Rate, Sec^{-1}		
		1st repl	2nd repl	3rd repl	1st repl	2nd repl	3rd repl
2.5	186	4.5	3.5	3.5	0.558	0.434	0.434
5	372	6.5	5.0	5.0	0.806	0.620	0.620
10	744	10.0	7.0	7.8	1.24	0.868	0.967
20	1490	14.0	9.5	11.5	1.74	1.18	1.43
50	3720	24.0	17.0	19.5	2.98	2.11	2.42
100	7440	33.5	27.0	33.0	4.15	3.35	4.09

TABLE XXV

SHEAR STRESS AND STRAIN RATE VALUES FOR 2.233.lit/kg AT 165°F; USING 4 MM GROUND CORN

		Dial Readings			Strain Rate, Sec^{-1}		
RPM	Shear Stress dyne/Cm^2	1st repl	2nd repl	3rd repl	1st repl	2nd repl	3rd repl
2.5	185	1.2	1.5	1.5	0.149	0.186	0.186
5	372	1.7	2.0	2.7	0.211	0.248	0.355
10	744	2.5	2.7	4.0	0.310	0.335	0.496
20	1490	3.7	4.0	5.7	0.459	0.496	0.707
50	3720	6.0	7.0	6.4	0.744	0.868	0.794
100	7440	10.0	11.0	9.8	1.24	1.36	1.22

TABLE XXVI

SHEAR STRESS AND STRAIN RATE VALUES FOR 2.233.lit/kg AT 200°F; USING 4 MM GROUND CORN

RPM	Shear Stress dyne/Cm^2	Dial Readings			Strain Rate, Sec^{-1}		
		1st repl	2nd repl	3rd repl	1st repl	2nd repl	3rd repl
2.5	186	1.5	1.8	2.5	0.186	0.223	0.310
5	372	2.5	2.5	4.0	0.310	0.310	0.496
10	744	4.5	3.5	5.2	0.558	0.434	0.645
20	1490	5.2	5.0	7.5	0.645	0.620	0.868
50	3720	8.0	8.5	10.0	0.992	1.05	1.24
100	7440	13.5	12.5	14.0	1.67	1.55	1.74

TABLE XXVII

SHEAR STRESS AND STRAIN RATE VALUES FOR 2.680.lit/kg AT 165°F; USING 4 MM GROUND CORN

RPM	Shear Stress dyne/Cm^2	Dial Readings 1st repl	2nd repl	3rd repl	Strain Rate, Sec^{-1} 1st repl	2nd repl	3rd repl
2.5	186	1.7	0.6	0.5	0.124	0.0744	0.062
5	372	1.2	0.8	0.9	0.149	0.0992	0.112
10	744	1.7	1.1	1.5	0.211	0.136	0.186
20	1490	2.2	1.8	1.7	0.273	0.223	0.211
50	3720	3.2	2.7	2.5	0.397	0.335	0.310
100	7440	4.3	4.5	4.0	0.558	0.533	0.496

TABLE XXVIII

SHEAR STRESS AND STRAIN RATE VALUES FOR 2.680.lit/kg AT 200°F; USING 4 MM GROUND CORN

RPM	Shear Stress dyne/Cm^2	Dial Readings 1st repl	Dial Readings 2nd repl	Dial Readings 3rd repl	Strain Rate, Sec^{-1} 1st repl	Strain Rate, Sec^{-1} 2nd repl	Strain Rate, Sec^{-1} 3rd repl
2.5	186	1.0	0.8	0.9	0.124	0.0992	0.112
5	372	1.5	1.0	1.2	0.186	0.124	0.149
10	744	2.0	1.3	1.7	0.248	0.161	0.211
20	1490	2.7	2.4	2.2	0.335	0.298	0.273
50	3720	4.3	4.0	3.5	0.533	0.496	0.434
100	7440	5.0	5.7	6.0	0.620	0.707	0.744

VITA

Ali Khakbaz

Candidate for the Degree of

Master of Science

Thesis: VISCOMETRIC PROPERTIES OF CORN WATER SLURRIES DURING COOKING FOR ETHANOL PRODUCTION

Major Field: Agricultural Engineering

Biographical:

Personal Data: Born in Tehran, Iran, January 25, 1958, the son of Ebrahim and Zahra Khakbaz.

Education: Graduated from Haddaf #1 High School, Tehran, Iran, in 1975; recieved the Bachelor of Science in Agricultural Engineering degree from Oklahoma State University in December 1979; completed requirements for Master of Science degree at Oklahoma State University in May 1982.

Professional Experience: Graduate Research Assistant in the Agricultural Engineering Department, Oklahoma State University, January 1980 to December 1981.

Professional Organizations: Student member of American Society of Agricultural Engineering, Oklahoma Society of Professional Engineers.