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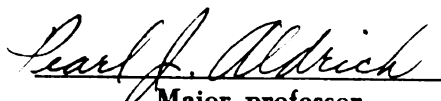
The Effect of Sucrose, Citric Acid, and Temperature
on the Viscosity and Gel Strength of
Cornstarch Pastes

presented by

Miss Ann Josephine Hains

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The Effect of Sucrose, Citric Acid, and Temperature
on the Viscosity and Gel Strength of Cornstarch Pastes

by

Ann Josephine Hains

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TABLE OF CONTENTS

	Page
I. ACKNOWLEDGEMENTS	1
II. INTRODUCTION	1
III. REVIEW OF LITERATURE	4
Chemical Fractions	4
Effect of Swelling on Physical Characteristics	6
Viscosity	8
Inherent Starch Characteristics	9
Treatment of Starches during Manufacture .	10
Concentration of Starch	12
Presence of Electrolytes	12
Effect of Time and Temperature	13
Effect of pH	16
Gel Properties	21
Effect of Sucrose	22
Objective Tests for Viscosity, Line Spread, Gel Strength	25
IV. PROCEDURE	36
Ingredients	36
Formulas	37
Treatments	40
V. RESULTS AND DISCUSSION	55

TABLE OF CONTENTS (Contd.)

	Page
VI. SUMMARY	79
VII. CONCLUSIONS	82
VIII. LITERATURE CITED	84
IX. APPENDIX	88

INTRODUCTION

The current trend in food production is toward simplification of recipes and methods of preparation. Some resistance is usually encountered when changes are made in practices which have long been accepted. One example of such resistance to change is found in the reluctance to accept modifications in the method of combining and cooking the ingredients of lemon pie filling.

A survey of pie filling recipes in large quantity cook books showed that generally the directions for making lemon pie filling recommend mixing all ingredients together and cooking this mixture until it thickens. In contrast, cooking procedures in most small quantity cook books follow the conventional method in which all the ingredients, except the egg yolk and lemon juice, are cooked together until thick. To this thickened starch paste the egg yolk is added and the mixture cooked long enough to coagulate the egg protein. After the completely cooked paste is removed from the heat, the lemon juice is added.

Several authors of textbooks on food preparation explain that the lemon juice is added to the cooked starch paste to minimize the hydrolyzing effect of the

acid on the starch and the subsequent thinning of the mixture. According to authorities on food preparation, the rate of hydrolysis increases with rise in the temperature of the pastes and with increased concentration of acid. This appears to offer a feasible explanation for the conventional method recommended for cooking lemon pie fillings. However, it fails to explain why methods, which are not based on principles of the theory of acid hydrolysis of starch, produce fillings which are as thick as those made by the conventional method.

The literature contains reports of a few experimental studies on the effect of sucrose on the thickening of cornstarch pastes containing relatively small proportions of starch (22, 36, 40). A limited amount of work on the effect of lemon juice on the viscosity and gel strength of cornstarch pastes has also been reported (8, 15, 36).

The first objective of this investigation is to study the effect of different concentrations of sucrose on the viscosity and gel strength of a cornstarch paste of constant composition. The second objective of this study is to determine the effect of specific amounts of citric acid on the same characteristics of the sweetened starch pastes. Since the proportions of acid and sugar used in this study fell within the range of those used in lemon pie fillings,

it is hoped that the results will be interesting and informative to those concerned with producing high quality foods by simplified methods.

REVIEW OF LITERATURE

Schoch (34), in his review of starch research, stated that the chemistry of starch has probably excited more interest over a greater period of years than has been elicited by any other single substance. Only since the late 1930's has real progress been made in explanation of the basic structure, composition, and behavior of starch. Modern colloid chemistry has facilitated this progress.

Chemical Fractions

In 1924, Alsborg and Rask (1) found that starches of certain cereals gave a red color with iodine and others gave a blue color. They stated that a more viscous paste was produced by the starches which gave a red color with iodine than by those which gave a blue color. In 1938, Woodruff and Mac Masters (39) performed a series of experiments with commercial and laboratory extracted cornstarches. They found a marked deviation between the viscosity readings and the gel strength in all the starch samples. They concluded, "apparently the two sets of measurements emphasize different points of behavior and both may be important." The following year,

Brimhall and Hixon (9), showed the same lack of correlation between the rigidity and viscosity values of 9 kinds of cornstarch. A few years later, Knowles and Harris (27) performed experiments on wheat starch. They stated that the lack of correlation between viscosity of the gelatinized wheat starch and its gel strength indicated that these were measurements of two different properties.

Starch can now be chemically fractionated into two components. Schoch (34) prefers to call these the A-fraction and the B-fraction. Other writers (24, 25, 27, 33, 37) prefer to the A-fraction as amylose and the B-fraction as amylopectin.

Amylose, which is composed of D-glucose units linked in linear chains with no branching, gives a blue color with iodine and is soluble in hot water. Its solutions show a great tendency to retrograde, that is, revert to an insoluble state on cooking. In a 5 per cent concentration of cornstarch, the amylose sets into a rigid, irreversible gel. The behavior of amylose is of practical importance in food preparation only when gel formation is desired. The most common starches, corn, wheat, potato, rice, and tapioca, contain from 15 to 30 per cent amylose. Whistler and Weatherwax (38), in an analysis of starch made from 39 unimproved varieties of corn collected from

New Mexico, Arizona, and South America, found the amylose content varied from 22.2 to 23.3 per cent and the starches made from 7 varieties of corn from the standard corn belt averaged 23 per cent amylose. The Western Regional Research Laboratory (34) found that starch from common wrinkled-seeded peas contains at least 75 per cent amylose.

Amylopectin is made up of highly branched molecules and gives a red to violet color with iodine. It is soluble in water at concentrations of 5 to 10 per cent and the solutions are relatively stable. Amylopectin is the most important constituent in starches used as thickening agents, protective colloids or emulsifiers, and as sizing for paper and textiles. The most common starches, previously listed, contain a major proportion of amylopectin. Waxy strains of many common cereals, except wheat, have been discovered. The starches from these cereals are made up almost entirely of amylopectin and contain from 0 to 6 per cent amylose (38).

Effect of Swelling on Physical Characteristics

The literature contains numerous references to the changes which take place in the starch granule during cooking. Alsberg and Rask (1) mentioned the swelling and rupture of starch granules. Brimhall and Nixon (9)

concluded that after the temperature of maximum viscosity was reached, the granules became wrinkled rather than enlarged, the degree of wrinkling increasing with the rise in temperature. They reported changes were not attributable to rupturing of the granule but to increased permeability of the membrane which then offered no resistance to exchange of contents with the outside medium. According to Anker and Geddes (2), granule disintegration was not an important factor until swelling had progressed to a point where granules became closely packed. At this time, internal shearing stresses increased to the extent of granule rupture. More recently, Bechtel (3) reported that during cooking a complex series of starch changes occurred, which included swelling, collapse of the granules, solution of some of the constituents, and more or less complete granule disintegration. About this time, Campbell and co-workers (13) substantiated that viscosity changes during heating were caused by swelling, rupture, and dispersion of starch. They added that swelling of starch suggested a mechanical explanation for rapid increase in viscosity which altered flow characteristics because of congestion of distended granules. As the granules ruptured, the starch was dispersed and the viscosity dropped. Schoch (34) suggested that the old theory of an envelope or sack enclosing the

starch granule appeared untenable and that, instead, the swollen granules must be regarded as gel particles held together by associative hydrogen bonding between branches of the B-fraction. In a review of the findings of many research workers, Kerr (26, p. 11) concluded that swollen granules slowly contract when maintained in water at temperatures above 50°C. Rupture of these distended granules may result from severe agitation, chemical treatment, or other methods of handling starch which involve high shearing stresses.

The term, gelatinize, is used to refer to the changes starch undergoes as it swells in water during cooking. Gel is defined as a jelly-like material formed by the coagulation of a colloidal liquid. Gelation is the term used to describe the cooling and solidifying of the cooked starch paste.

Viscosity

In the literature on starch research the term "apparent viscosity" is used frequently (2, 6, 17). It is defined as the shear-dependent viscosity and is established by the ratio of shearing stress to the rate of shear. According to Caesar (11), and

to Bechtel and Fischer (6), consistency and viscosity can be used interchangeably. Sjostrom (35) decided that fluidity or viscosity in a paste is not the same as in a homogeneous liquid. In the paste, viscosity is caused largely by a suspension of both the whole and the disintegrated granules, and the degree of viscosity is a measure of the extent to which swollen particles crowd each other in passing.

Some of the variables which may affect the viscosity of a starch paste include the inherent starch characteristics, treatment of starches during manufacture, mechanical injury of the particles, concentration of the starch, presence of materials other than starch, time and temperature of cooking, and pH of the cooking medium.

Inherent Starch Characteristics

It is well known that the inherent starch characteristics affect the action of the granules during cooking and during cooling. This has been mentioned previously in the discussion on chemical fractions. In their work on wheat and corn starches, Woodruff and Mac Masters (39) stated that some of these starches produce different pasting results, a fact which may be of practical

significance to starch users. Mangel and Bailey (29) found that hard spring, hard winter, soft winter, and durum wheat gave different results. Their conclusion was that chemical differences, which cause differences in physical properties, are probably complex in nature and may be attributed to morphological differences in the starch granule. In a comparison of various kinds of commercial starches, Nutting (31) found that potato starch swelled most and gave the most viscous, unstable, and variable paste. The experiments of Harris and Jespersen (31) with wheat, barley, and corn starches verified the above finding. They reported a significant difference in the swelling power among these kinds of starches.

Treatment of Starches during Manufacture

The effect of treatment of starch during manufacture has received much attention from research workers. Woodruff and Mac Masters (32) prepared their own cornstarches and made comparisons with commercial products. They discovered measurable differences in the cooking properties between the starches extracted by various methods. The experiments by Caesar (11) on three commercial cornstarches indicated

variation in their swelling power and stability. Two samples thickened more quickly and to a greater degree than the third, which thickened less but also thinned more slowly and to a lesser degree than the others. In a discussion of starch characteristics, Sjostrom (35) said, "thin-boiling indicates these starches give a paste which is more fluid than that of ordinary starch, and by suitable modification of treatment varying degrees of fluidity can be obtained". Campbell and co-workers (13) emphasized the need for selecting a starch which will produce the results desired in cooking various food products. They commented that chemical and heat treatments in production may affect starch pasting characteristics. They pointed out that drying of starch during manufacture may result in a slight gelatinization which increases the ability of starch to absorb or hold water and, therefore, increases the viscosity of a paste made with the starch.

Bechtel (8) verified these facts and pointed out that various samples of the same designation may give different results if they have been prepared under different conditions of manufacture. After studying laboratory extraction of wheat starches, Harris and Jespersen (21) concluded that it is useless to report data on starch research unless the technique of manufacture is included also.

Concentration of Starch

The experiments of Anker and Geddes (2) showed that as the concentration of starch was increased there was an appreciable decrease in the temperature of the first measurable change in viscosity. There was also a marked increase in maximum viscosity and a slight decrease in the paste temperature at maximum viscosity. In addition, the point of greatest consistency was more abrupt and the subsequent decrease more rapid. Besides substantiating these findings, Fechtel and Fischer (6) and Brimhall and Hixon (9) established the fact that beyond the point of a critical concentration of starch, each increase made enormous increases in viscosity and rigidity of resulting pastes. Fechtel (3) and Lowe (33) reported that the extent of breakdown of starch pastes, after they had reached maximum viscosity, was greater in the more concentrated pastes.

Presence of Electrolytes

Distilled water was used by Fechtel (5) in making tests on starch, because he found that the presence of electrolytes affected the final results. Richardson and Higginbotham (32) reported that starch had greater

swelling power in distilled water than in the presence of any salts. Distilled water was used in the work reported by Woodruff and Mac Masters (39) and by Harris and Jespersen (20). Morse and his co-workers (30) used varying concentrations of dry milk solids and starch with both tap water and distilled water in their experiments. They concluded that there was no appreciable difference in gel strength of thick pastes made with either tap water or distilled water. However, with thin pastes they found that viscosity was greater with distilled water than with tap water. Bisno (8) stated that periodic variations in the water supply were sufficient to result in significant differences in the consistency of pie fillings and that stability, viscosity, and tendency to gel varied with different water supplies. He added that a change from tap water to distilled water or softened water frequently resulted in noticeable changes in consistency of pie fillings.

Effect of Time and Temperature

The Stormer Viscometer (p. 27) was used by Alsberg and Rask (1) to study pastes of 4.5 to 5 per cent concentration of commercial cornstarch in a water medium. They

reported that there were no significant changes up to 65°C. The initial viscosity rise began between 65° and 68°C; the viscosity rose gradually from this point until it reached a maximum at 91°C. They concluded that simultaneous and gradual changes occur over a span between these temperatures and that gelatinization temperature has no sharp transition point. These findings were substantiated by Caesar (11), who suggested that the temperature of the initial change be accepted as the gelatinization point of starch under given conditions of cooking and specific concentration. In addition, Caesar (11) found that the consistency changes in starch and water pastes comprise the four phases: (1) starch and water suspension, (2) gelatinization of starch granules or thickening, (3) partial rupture of starch cells or thinning, (4) association or rethickening upon cooling. Although starches may vary in the time-temperature viscosity curve that they make, most of them have similar curves. Caesar and Moore (12), Bechtel (3), and Grimhall and Hixon (9), and many others have verified this starch behavior.

Both time and temperature affect the viscosity of starch pastes. According to Bechtel (3), rapid heating lowered the temperature of the initial rise in viscosity, decreased the time at which starch pastes needed to be held

at 90°C to reach maximum consistency, and increased the viscosity of the pastes. When the water bath temperature was raised sufficiently to produce pastes at 92° to 94°C, the rate of heating of the pastes increased slightly with no observable effect on the curve of viscosity rise.

However, continued cooking at the higher temperature caused a greater breakdown in paste consistency. This decrease in viscosity was intensified by agitation.

Bechtel (3) stated that when starch pastes are stirred during cooking, a difference of 2°C in bath temperature produced less than 1°C change in the temperature of the final paste but produced noticeable changes in paste properties. He concluded that there appeared to be a specific temperature at which each type of starch gelatinized at a steady rate and at which it could be held for a reasonable time without excessive breakdown of viscosity. For unmodified cornstarch, this temperature was about 90°C. These findings have been supported by Anker and Geddes (2) and by Lowe (23). Campbell and co-workers (13) stressed the importance of knowing the effect of the time and temperature on the selected starch if one desired to produce uniform puddings or pie fillings. According to Eisno (8), pie fillings for commercial production should be heated rapidly to the optimum temperature,

removed immediately from the cooking kettle, and cooled quickly to produce maximum viscosity.

In most of the starch research, the water used in making the starch slurry was at room temperature. However, Anker and Geddes (2) made starch slurries with water at 45°C and above this temperature. They found that applying heat to these slurries produced a marked increase in the maximum paste viscosity. They also stated that mixing and holding the suspension at room temperature for 15 minutes before cooking increased the initial gelatinization temperature and produced a more viscous paste than was obtained in pastes which were cooked immediately after mixing the slurries.

Effect of pH

It is generally agreed that the pH of the starch suspension affects its cooking properties. However, the literature does not give a clear-cut picture of these effects. As mentioned earlier, some investigators extracted the starches which they used in preference to commercial products. Residues of acids and alkalies used in the manufacture of commercially prepared starches introduced variables which made controlled studies

impossible, they felt. Most of the experiments in starch research have been done with inorganic acids. Bechtel (3) adjusted the pH of the cornstarch slurries with HCl and NaOH. He concluded that unmodified cornstarch showed no breakdown between the pH values of 4 and 7 after 20 minutes cooking and that a given degree of alkalinity caused a breakdown of paste viscosity. The pH of wheat starch suspensions was adjusted with buffers by Anker and Geddes (2). They found that there was a decrease in maximum paste viscosity with increased pH. However, the control sample which had a lower pH gave a greater peak viscosity than those which were buffered at higher values. They suggested that these results may have been caused by the effect of ion adsorption on the granules.

Harris and Banasik (19) studied the effect of acids and alkalies on the cooking of one commercial cornstarch and several laboratory extracted wheat starches. They reported that cooking starches in the acid medium increased granule swelling at temperatures of 90°C and above. NaOH increased granule swelling at 70°C and above, but the increase was smaller at 95°C than that obtained with HCl. The work done by Morse and his associates (30) with the dry milk solids and starch pastes showed that the addition of vinegar, sufficient to bring the pastes to a pH 6,

caused the mixture to curdle, which made accurate viscosity readings impossible. The pastes were cooled and were found to have an appreciable decrease in gel strength when compared with similar gels which were not treated with vinegar. Chapman and Buchanan (14) did many experiments with cornstarch pastes and various inorganic acids. They concluded that addition of acids in concentrations as low as 0.0025 N caused a decrease in viscosity and that the addition of dilute acids to the starch and water suspensions prevented the formation of gels.

According to Trempel (36), most pie fillings contain varying amounts of acid which gradually destroy ordinary starches and cause these starches to lose their water holding ability. To decrease this hydrolyzing effect of acid, he recommended that the cooking time be reduced to a minimum. Trempel added that bakers often cut the cooking time so much that the starch is not cooked thoroughly or that they overcook the product and destroy some of the starch. Recently, Bisno (8) stated that cooked cornstarch pastes may become thinner on holding after preparation and suggested that this change in viscosity may be caused by rupturing of the starch granules or by the conversion of starch to dextrin, maltose, and dextrose. He added that this conversion, which is accentuated by acid and

heat, may result in a rapid thinning when lemon juice has been added to cooked filling and the hot mixture is allowed to stand for several hours. Bisno also reported that eggs or other buffering material could counteract this thinning effect. Lowe (28) supported the theory that acid caused a hydrolysis of the starch molecule and, that stiffening power decreased as hydrolysis proceeded. She stated that workers have generally agreed that acid hydrolysis is more rapid with increased paste temperature, longer holding periods at the higher temperature, and increased acid concentration. Lowe did not state the kind of acid or the amount necessary to produce this hydrolysis.

In a report of recent work, Kerr (29) said that it has been presumed that modification of starch with acid is the result of a limited hydrolysis of the carbohydrate molecules during which the high polymer is split into molecules of progressively smaller size as the action of the acid continues. However, the result of investigations could indicate that, under certain conditions, when acid begins to work on cornstarch, the straight chains are protected by the formation of a resistant complex in which amylose and particles of amylopectin combine. The branched amylopectin molecules are the first to be split and this disintegration and weakening of the granule

structure may explain why pastes made with acid-treated starch show a great reduction in consistency. This same low viscosity starch, cooked in a relatively small proportion of water, may have a very high gelation power despite its extensive degradation as reflected by its low viscosity.

Chidester (18) extracted cornstarch from yellow cornmeal. She cooked 6 per cent cornstarch pastes and repeated this cooking with the substitution of lemon juice for part of the liquid. From subjective observation, she reported that the gels of pastes cooked with lemon juice seemed stronger and that the gels of pastes to which lemon juice was added at the end of the cooking period were definitely stronger than similar pastes prepared without the addition of juice. Chidester continued that the addition of lemon juice to 6 per cent cornstarch pastes, which were sweetened with 1.9 per cent sucrose, brought about an increase of gel strength. In pastes made with the same proportion of cornstarch and 25.8 per cent of sucrose, the addition of lemon juice caused formerly unmovable pastes to form a gel which almost held the shape of the mold. Lemon pie fillings, made by varying constituents, were studied by Chidester (18). She stated that the lemon juice

counteracted the gel weakening effect of the sugar. She also reported that the juice was more effective in forming a strong gel and in controlling syneresis when it was added at the end of the cooking period rather than with the egg yolk in the second stage of cooking the mixture.

Gel Properties

In this review, some mention has been made of the gelation of cooled starch pastes. Additional studies have been made which emphasize the gel properties of starch. Bechtel (5) heated unmodified cornstarch suspensions to 91°C and then cooled them in a water bath held at 25°C . Some gels were tested at the end of 4 hours and others at the end of 24 hours. He found that the rigidity and breaking strength increased most during the first few hours and that the increase after that time became progressively slower. The gel strength of pastes cooked to 94°C was greater than the gel strength of pastes cooked at lower temperatures but decreased if cooked beyond 94°C . Additional tests, in which the slurries were brought to pH 6 with HCl, were made on acid-modified cornstarches. These showed that the rigidity and breaking strength did not decrease to the same extent

that the viscosity of these pastes decreased. Wheat starch was used for the experiments of Knowles and Harris (27). They found that 6 per cent starch suspensions in a water medium produced firm gels. The critical gel formation temperature was 90°C , and above this temperature the gels were firm. Below this point the gels were weak and showed syneresis after standing for 10 hours. Woodruff and Nicoli (40) did some studies with starch gelation and used photographs to illustrate their findings. They concluded that 5 per cent commercial cornstarch pastes cooked to 87°C produced very weak, granular gels. Some samples were heated for 15 and for 30 minutes at this temperature but there was little improvement in gelation. Heating to 90° , 95° , and 99.5°C gave gels which were indistinguishable from each other.

Effect of Sucrose

Further work by Woodruff and Nicoli (40) was done with 5 per cent suspensions of corn, wheat, rice, potato, and cassava starches and varied amounts of sucrose. With the addition of 10 or 30 per cent sucrose, it was found that the root starches formed increasingly softer gels. The addition of 50 or 60 per cent sugar produced a syrup

instead of a gel. When the same percentages of sucrose were added to the cereal starches, there was an increase in transparency and tenderness with each increase of sugar. The addition of 50 per cent sucrose produced a gel that would not hold the shape of the mold, and the addition of 60 per cent sucrose made a syrup with the cereal starch pastes. All the samples in the above tests were left in the molds for 24 hours before they were checked for firmness and were photographed.

Hester (22) reported some comparative studies of physical properties of pastes made from cornstarch, soft wheat starch, soft wheat flour, hard wheat starch, and hard wheat flour. She used the Brabender amylograph and the Brookfield viscometer for viscosity measurements, and the line spread device, described by Grawemeyer and Pfund (18), for measurement of the spread of pastes. Gels were aged and then measured for rigidity by an Exchange Ridgelimeter and for elasticity and breaking point by an embedded-disk method, which was similar to that described by Saare and Martens and modified by Kerr (26, p. 135-137). In her research, Hester used 6.5 per cent cornstarch in distilled water for the basic starch paste. She reported the effect of 15.9, 23.7, 31.6, and 39.5 grams of sucrose per 100 grams of water on viscosity, spread, rigidity,

elasticity, and breaking point of the starch pastes. She concluded that increasing concentrations of sucrose increased the temperature of the initial viscosity rise and the temperature of the maximum viscosity of the cornstarch pastes. With the lowest sucrose addition, 15.9 grams, she found that the peak of the viscosity curve was higher than that reached by the cornstarch and water paste. However, the increased quantities of sucrose, beyond the 15.9 gram concentration, resulted in a progressive decrease in viscosity of the cornstarch paste.

In the tests on gel strength, Hester (22) found that the gels of the 6.5 per cent cornstarch paste plus 15.9 grams of sucrose per 100 grams of water were slightly less rigid than those of similar pastes without sucrose. With a concentration of 23.7 grams of sucrose, the gels retained their form for 30 to 60 seconds and then collapsed. With sucrose concentrations of 31.6 grams and 39.5 grams in the basic cornstarch paste, thin pastes instead of gels resulted.

Trempel (36) stated that the addition of sugar raised the gelatinization temperature of a starch and water suspension. He continued that the addition of seven to eight pounds of sugar to one pound of cornstarch in a specified

amount of water resulted in incomplete cooking of the starch under ordinary cooking conditions. The resulting cooled gel was dull, thin, and had a raw starch taste. Trempel said that the proportion of sucrose should not be greater than three and one-half times the amount of starch used. For the best results in cooking pie fillings, he concluded that the weight of the sugar to be cooked as a part of the paste should not exceed twice the weight of the starch. Additional sugar required for flavor should be added to the hot, cooked product.

Although many research workers agree upon some of the physical properties of starch pastes, others report conflicting results. Much more investigation is needed for better understanding of the effect on the behavior of the starch granule of additions of other materials to starch and water pastes.

Objective Tests for Viscosity, Line Spread, and Gel Strength

Some of the characteristics of starch pastes are viscosity, plasticity, gel strength, and rigidity. Functional properties of starch pastes are determined by tests of the physical properties of the pastes.

Viscosity measurements

Caesar consistometer. This is a continuous recording apparatus which measures several characteristics of starch pastes simultaneously (13). The starch slurry is placed in a beaker which is surrounded by a heating bath, the temperature of which is raised at a regulated rate. A paddle, connected by a shaft to an electric motor, is suspended in the beaker. The motor and paddle move at constant speeds, and the changes in viscosity are indicated by the differences in the electrical input to the motor necessary to maintain a constant speed of stirring. After the starch is cooked, cold water may be introduced into the bath and the characteristics of the cold paste may be determined.

This apparatus is reported to be particularly well adapted to studies of pastes with relatively high concentrations of starch. Caesar and Moore (13) reported that a concentration of 20 per cent starch is easily recorded by this instrument. The continuous history of pasting obtained in this test includes the gelatinization point, the peak viscosity with a definite cooking procedure, specific viscosity after a prescribed cooking operation, rate of increase in paste body with decreased temperature,

and cold paste body after a specified time interval at the lower temperature.

Brookfield viscometer. This torsional type of viscometer (22) contains spindles of various capacities which range from 1 to 1000 poises. These spindles are driven by a synchronous motor. The viscosity measurements of the cooked paste are made at different rates of shear and are expressed in terms of poises.

Stormer viscometer. The Stormer viscometer is used for determining the viscosity of a cold starch paste. The starch is gelatinized and cooked for 15 minutes. The paste is placed in a closed container and held in a constant temperature bath, preferably at 25°C. At the end of the aging period, the surface skin is removed and discarded. The remaining paste is stirred lightly and transferred to the cup of the Stormer viscometer. The apparatus has a cylinder which is immersed in the test paste and is rotated by a free-falling weight acting through a gear-and-pulley system. The time in seconds necessary for a given weight to produce a certain number of revolutions is taken as a measurement of cold paste body (20, p. 193).

Brabender amylograph. In this instrument the starch slurry is placed in a cup which is surrounded by an air bath for temperature control. The cup is revolved at a constant speed. The measuring and stirring device consists of a disc to which are attached several short rods extending into the paste. The torque impressed on the measuring unit is transmitted to a recording torsion balance and a continuous graphic record is traced by the instrument over the entire period of the test. The temperature may be increased at a fixed rate and may be held at any level by a thermostatic control. By switching off the heating element, the paste may be cooled to any desired temperature. In addition, a cooling coil is provided for the circulation of cold water to accelerate cooling of the paste for measuring consistency changes brought about by rapid reduction of temperature (26, p. 127).

Corn Industries viscometer. In this instrument (3, 4), the viscosity of the starch paste is determined and recorded continuously for any desired cooking period. The apparatus can be adjusted for a cooling period if desired. A steel beaker is placed in a thermostatically controlled liquid bath. Distilled water is used in the bath unless cooking at the boiling point of water is desired. In such cases, glycerol, Prestone, or other

Liquid with a boiling point above 100°C is used. The cooking paste is stirred by electrically driven paddles at a constant rate. The stirring device, of unique design, removes the layer of pasted starch from the walls of the beaker. This constant removal of the pasted layer improves the efficiency of heat transfer to the body of the paste and results in a nearly uniform paste temperature. A separately mounted propeller, driven through a gear differential, stirs the center of the paste and measures the viscosity changes. The force which the propeller encounters is continuously balanced by a dynamometer consisting of a weight arm which moves through a vertical arc. A series of interchangeable weights are provided for the dynamometer so that it covers a relatively wide range of torques from 250 to 2000 gram-centimeters to attain equal sensitivity with dilute and concentrated pastes. Attached to the dynamometer is the pen which automatically records the viscosity changes of the paste. Evaporation of moisture from the paste during cooking is prevented by a condenser built into the cover of the beaker.

The usual technique of operation consists of preheating the bath to the specified temperature. The motor-driven stirrer is then started. The starch slurry, made

from water at room temperature, is mixed and poured into the steel beaker. The recorder is started and the condenser cover is put in place. From this point the test is automatically controlled except for the reading and recording of the paste temperature at the desired intervals.

Scott test. The Scott test is a widely used method for determining hot paste viscosity. A quantity of starch at known pH is stirred to a slurry with 280 cubic centimeters of distilled water in a German-silver beaker. The beaker and contents are then placed in a boiling water bath and heated, with stirring, until the starch is gelatinized. The complete cooking period is 15 minutes, but at a definite period of time prior to this 15 minutes, 200 cubic centimeters of the paste are transferred to the viscosity cup of the Scott apparatus. This cup is also placed in a boiling water bath. At the end of a total heating time of 15 minutes, the plunger valve which closes the orifice on the bottom of the cup is raised and the time in seconds is noted for a given volume of the paste to fall into a graduated cylinder. The time, in seconds, is the specific Scott test viscosity.

This viscosity may be considered as a relative viscosity in the sense that standard starches are used to set up permissible limits of variation in the viscosity of other starches to be tested by this method (26, p. 119-121).

Line spread tests

Grawemeyer and Pfund test. Grawemeyer and Pfund (13) used a line-spread test for observing the spread of applesauce and cream pie fillings. In this test a flat, glass plate was placed on a surface checked for evenness with a spirit level. Beneath the glass plate was placed a diagram of concentric circles. The diameter of the smallest circle was two inches and diameters of surrounding circles were graduated in one-eighth inch intervals. A hollow cylinder of the exact diameter of the innermost circle was needed for holding the paste sample. For this purpose Grawemeyer and Pfund (13) used a biscuit cutter from which the handle was removed. The cylinder was placed on the glass plate directly over the smallest circle, filled with the material to be tested, and leveled with a spatula. The cylinder was lifted carefully and the material allowed to spread for two minutes, and readings were taken at four equally spaced radii on the diagram. The average of the four readings represented the number of one-eighth inch units a given volume of material at room temperature had spread in two minutes.

Billings modification of spread test. Billings (7) used a modification of the Grawemeyer and Pfund method for the

radius of spread of hot pie fillings. The line spread mat she used was a 25.4 centimeter square brass plate, sprayed with Etanol which gave the mat a permanent black finish. This mat was etched with eight concentric circles, one centimeter apart. Between the circles the mat was marked at two millimeter intervals at four equi-distant radii. The brass plate was kept hot during the period of testing by placing it over a pan of boiling water. A hollow brass cylinder, the same diameter as the zero circle was placed on the mat and filled with the hot pie filling. The filling was levelled with a spatula and the cylinder was carefully lifted by means of a test tube holder. The hot material was allowed to flow for 30 seconds and the spread was read on each of the equally spaced radii on the mat. The radius of spread was determined by averaging the four readings. Comparative readings for the cold fillings were made using the same mat unheated.

Gel strength tests

Exchange Ridselimeter. The Exchange Ridselimeter is described by Cox and Higby (10). For this test, the pastes are stored in jelly glasses provided with sideboards. For testing the paste, the sideboards are removed

and the excess gel is sliced off to the rim of the glass. Each gel is removed by inverting the container on a glass plate. After the gel has stood on the plate for two minutes, a micrometer screw is adjusted and the per cent of sag is read directly. This test measures the rigidity of a gel.

Tarr-Baker jelly tester. The breaking strength of a gel can be tested by the Tarr-Baker jelly tester (36, p. 134-135). The paste is placed in a porcelain cup, covered with a film of light mineral oil, and aged in a water bath at 20°C for one hour. For this test, the oil is drained off and the dish of gel is placed under the plunger of the tester. With 500 cubic centimeters of water in the Tarr-Baker testing jar at the start, the flow of water is adjusted so that it rises in the manometer column 60 centimeters per minute. The manometer is read when the gel breaks. The gel strength is reported as the height in centimeters reached by the water in the manometer at the breaking point of the gel. Tests are made in triplicate and averages are made for the three readings.

Kerr modification of Saare and Martens test. Among the first tests proposed for the evaluation of gel strength of a cold starch paste was that of Saare and Martens.

This method, modified by Kerr (26, p. 136-137), is used frequently. In this test, the starch paste is poured into a glass container to a specified level immediately after cooking. A circular metal disc of known diameter is suspended in the paste by means of a metal rod connected to the center of the upper surface of the disc. The metal rod, crooked at the top, is hung over a bar which rests on the top edges of the vessel holding the paste. The length of the rod is such that, when suspended over a horizontal bar, it permits the test disc to be immersed in the paste to a depth of three centimeters. A thin film of oil is placed on top of the starch paste and the vessel is held in a constant temperature bath at 25°C for 24 hours. Then the test container is placed on a bridge over one pan of a large, sensitive beam balance. A specially made hook, suspended from the beam of the balance, connects with the bend in the rod connected to the disc. Small size shot are added at a fixed rate to the other pan of the balance. The weight of the shot is noted at the time when the disc fractures the gel. The weight of the shot, less the weight of the disc and all connections to the beam of the balance, divided by the exact area of the lower surface of the disc is taken as the gel strength. The result is expressed in grams per square centimeter.

Penetrometers and gelometers

Fuchs penetrometer. According to Kerr (20, p. 137), most of the penetrometers and gelometers are not sufficiently sensitive to distinguish between various grades of modified industrial starches. However, Kerr states that the instrument constructed by F. W. Fuchs is sensitive and accurate. The plunger in this penetrometer is a highly polished, sharpened, hollow metal tube which resembles a cork borer. This plunger is attached to the bottom of a rod which is held in a true vertical position by several guides. At the top of the rod is a platform on which a weight is placed. After the starch paste is cooked, it is poured into a container which has a greater diameter than the plunger. After the paste sample has been aged for the desired period of time, approximately one-fourth inch is cut from the top surface of the gel and discarded. The plunger of the instrument, adjusted so it rests on top of the surface of the gel, is released. The time, in seconds, required for the hollow tube plunger to cut into the gel to a specified depth is noted.

PROCEDURE

Ingredients

Distilled water was used in making all starch slurries in this study.

The cornstarch for this project was supplied by the Corn Products Refining Company of Argo, Illinois, and the lot used was taken from a regular run of their commercial starch prepared for food uses. This starch had a Scott viscosity of 76 which is considered standard for this type of starch.

The entire lot of cornstarch was placed in polyethylene bags and was kept in a covered, stainless steel bin at room temperature.

Two lots of superfine granulated sugar were obtained from the College Food Stores. These two samples were mixed together and were kept in a covered stainless steel bin.

Citric acid, C. F. crystalline form containing one molecule of water, was obtained from Merck and Company, Incorporated. Composition of the citric acid used was as follows:

Oxalates	.050%
Phosphates	.001%
Sulphates	.002%
Iron	.0005%
Residue on Ignition	.015%
Insoluble	.005%
Water	1 molecule
Molecular weight	210.14
A.S.C. Specification	

Formulas

Starch concentration. The percentage of cornstarch in recipes for lemon pie was found to vary from 9 to 13.6 per cent of the liquid weight. The investigator selected 12 per cent cornstarch concentration for this study after preliminary tests with 5, 10, 11, 12, and 13 per cent cornstarch pastes. In these tests, it was found that the 13 per cent cornstarch pastes were too heavy for the instrument which was used to record viscosity. The proportion of cornstarch to distilled water was kept constant in the starch slurries in all replications.

Sucrose concentration. In recipes for lemon pie fillings which were checked sugar constituted 40 to 55 per cent of the weight of the liquid. For Series A of this study, the arbitrarily selected concentrations of sucrose included a control paste with no sugar, and three sweetened pastes in which sugar constituted 20, 40, and 60 per cent of the weight of the liquid. The weights of starch, sugar, and water, based on the desired percentage relationships, were calculated to yield a total volume of one quart because the beaker of the viscometer was designed to cook and mix this amount of material. This volume of cooked mixture was also needed for other tests on the pastes.

Table 1 lists the ingredients and the quantities of each used for Series A. Three replications of each variation were made in this series.

Table 1. Formulas for Series A.

Variation	<u>Cornstarch</u>		<u>Sucrose</u>		<u>Water</u>	<u>Total Volume</u>
	Gm.	%	Gm.	%	ml.	ml.
I	138.0	18	---	--	839	977.0
II	97.8	18	163	28	816	977.0
III	88.8	18	298	48	740	976.6
IV	81.0	18	435	60	675	976.0

Acid concentration. To study the effect of acid on the viscosity and gel qualities of sweetened cornstarch pastes, each variation of Series A was repeated with the addition of citric acid at three specified levels.

Lemon juice is reported to have a pH value of 2.0 to 2.4 and a 0.1 N citric acid solution to have a pH value of 2.2 (25). For preliminary testing, the contents of two 14 oz. cans of frozen lemon juice,¹ mixed together and tested with a Beckman pH meter, Model G, were found to have a pH value of 2.32.

¹Coastal Brand Lemon Juice, Ventura Coastal Company, Ventura, California.

To prepare a citric acid solution approximating the pH of lemon juice, the amount of citric acid crystals needed to produce a 0.1 N solution was calculated as follows:

$$\frac{\text{Molecular weight citric acid} \cdot 1 \text{ molecule water} \times \text{N of solution}}{\text{Number of dissociable H}^+}$$

$$\frac{192.14 \cdot 18 \times 0.1}{3} = 7.004 \text{ grams citric acid crystals per liter of distilled water.}$$

In preliminary testing the pH value of the 0.1 N citric acid solution was found to be 2.32.

Since the amount of lemon juice in large quantity lemon pie recipes was found to vary greatly, the amount of 0.1 N citric acid solution for Series B was arbitrarily set to correspond with recipes of high lemon juice content. Proportions of the ingredients used in this series, which was considered comparable in acid content to an acceptable lemon pie recipe, are shown in Tables 2 and 3. The pastes in

Table 2. pH determinations of formulas used in Series B with citric acid and lemon juice.

Vari- ation	Distilled Water ml.	Corn- starch gm.	Sucrose gm.	Acid Solutions ml.	pH Values of Slurries	
					0.1 N Citric Acid	frozen Lemon Juice
V	624	108.0	0	276	2.65	2.65
VI	565	97.8	163	250	2.63	2.43
VII	513	88.8	296	237	2.61	2.41
VIII	463	81.0	405	207	2.61	2.44

this series contained a 0.1 N citric acid solution in an amount representing 44.5 per cent of the water in the formulas.

The pH determinations of the starch-water and the starch-water-sucrose slurries, in the proportions used in Series B, were made with 0.1 N citric acid and also with the frozen lemon juice mixture. The results of these determinations appear in Table 2.

To study further the effect of different acid concentrations on the viscosity and gel qualities of sweetened cornstarch pastes, each variation of Series B was repeated with decreased and with increased amounts of the 0.1 N citric acid.

In Series C the 0.1 N citric acid constituted 40 per cent of the total liquid, and in Series D it constituted 49 per cent of the total liquid. The formulas used in Series B, C, and D are listed in Table 3.

Three replications were made of each variation in Series B, C, and D.

Treatment

Preparation of citric acid solution. The citric acid crystals were weighed on a analytical balance¹ in packets

¹Christian Becker, New York. Ohausomatic Balance.

Table 3. Formulas for Series C, E, D.

Series Identi- fication	Vari- ation	Starch gm.	Water ml.	Sucrose gm.	0.1 N Citric Acid ml.	Total Volume ml.
C 0.1 N Citric Acid 40.0%	IX	108.0	652	---	248	977
	X	97.8	690	163	249	977
	XI	88.8	536	296	264	978.6
	XII	81.0	489	408	186	976
E 0.1 N Citric Acid 44.5%	V	108.0	624	---	276	977
	VI	97.8	668	163	250	977
	VII	88.8	513	296	227	978.6
	VIII	81.0	468	408	207	976
D 0.1 N Citric Acid 45.0%	XIII	108.0	596	---	304	977
	XIV	97.8	640	163	275	977
	XV	88.8	490	296	250	978.6
	XVI	81.0	447	408	228	976

of 7.004 grams, the amount calculated to make 1 liter of 0.1 N citric acid solution. The crystals were wrapped in glazed weighing paper¹ and then in several layers of cellophane and were sealed with Scotch tape.

For preliminary testing, the pH value was determined for the 0.1 N citric acid solution immediately after the solution was made and also after it had been kept at room temperature for 18 hours. In both instances the pH value was 2.30. In this study, the citric acid solution was mixed in one liter quantities.

Preparation of slurries. The starch and sucrose were weighed on a Torsion Balance. The quantities needed for each series were weighed prior to making the tests but were not mixed together until they were to be used. A rubber scraper was used to remove the starch from the balance pan.

The citric acid solution and the distilled water were measured in 500 ml. and 100 ml. graduated cylinders. The slurries were mixed with a narrow rubber scraper in one liter glass beakers.

It is essential that the slurry be mixed until it is free from lumps. Because the behavior of the starch-water

¹Sargent Laboratory Supplies, E. I. Sargent and Company, Chicago. No. S-65222 Black.

mixture differed from that of the starch-sucrose-water mixtures, the procedure of mixing the slurries was different.

For the variations without sugar, the starch was placed in the glass beaker. Approximately three-quarters of the liquid was added and the mixture was stirred vigorously with a narrow rubber scraper until it was free from lumps. The slurry was poured into the steel cooking beaker. The remaining liquid was used to rinse the glass beaker and was then poured into the cooking container.

It was more difficult to obtain slurries free from lumps with the sucrose variations. The starch and sugar were placed in the beaker and were blended thoroughly in the dry state. Only enough liquid was added to moisten the ingredients and the mixture was stirred vigorously. With each increase of sucrose, the first liquid addition was gradually decreased. When the slurry was free from lumps, all but one-fourth of the liquid was added. The remaining liquid was used to rinse the glass beaker and the rinsings were added to the mixture.

It has been reported that the length of time a starch slurry stands before cooking affects the viscosity of the cooked paste. Therefore, it is essential that the mixing and rinsing procedures be done quickly and that the mixing time be the same for each replication.

Cooking procedure and viscosity tests. The Corn Industries viscometer, described in the review of literature, was used for cooking and measuring the viscosity of the pastes.

Figure 1 shows a cross section diagram of the viscometer.

Before the tests were made, the steel beaker, stirrer, and condenser cover were placed in the viscometer. The heating element was turned on. When the temperature inside the beaker reached 70°C , the motor which controlled the stirrer was started. Then the slurry was mixed, one half of the condenser cover was removed, and the slurry was poured into the cooking beaker. Immediately the recorder was started and the beaker cover was replaced.

The Corn Industries viscometer has a liquid heating bath which is thermostatically controlled. A thermometer was inserted into the bath. The thermostat was adjusted to obtain the desired bath temperature which was kept constant throughout the cooking series. Distilled water is used for the liquid in the bath unless a temperature of 100°C for the bath is desired. In this case, it is advisable to use Prestone or glycerol to avoid steaming and excessive evaporation from the bath. Since this study was done with a 100°C bath temperature, Prestone was used for the liquid medium.

Figure 1. Sectional drawing of the Corn Industries
Viscometer (21, p. 133).

1. Recorder and dynamometer (Dynamometer not shown)
2. Cable from viscometer to recorder
3. Cable drum
- 4, 5, 6, 7. Gears of sun and planet differential
8. Worm, turned by synchronous motor (not shown)
9. Worm gear
10. Spring pins for holding center shaft
11. Coupling to attach stirrer
12. Condenser and cover
13. Liquid bath
14. Overflow
15. Drain cock
16. Starch beaker
17. Electric heater, thermostatically controlled
18. Scraper blades
19. Propeller
20. Thermometer in paste
21. Thermometer in bath

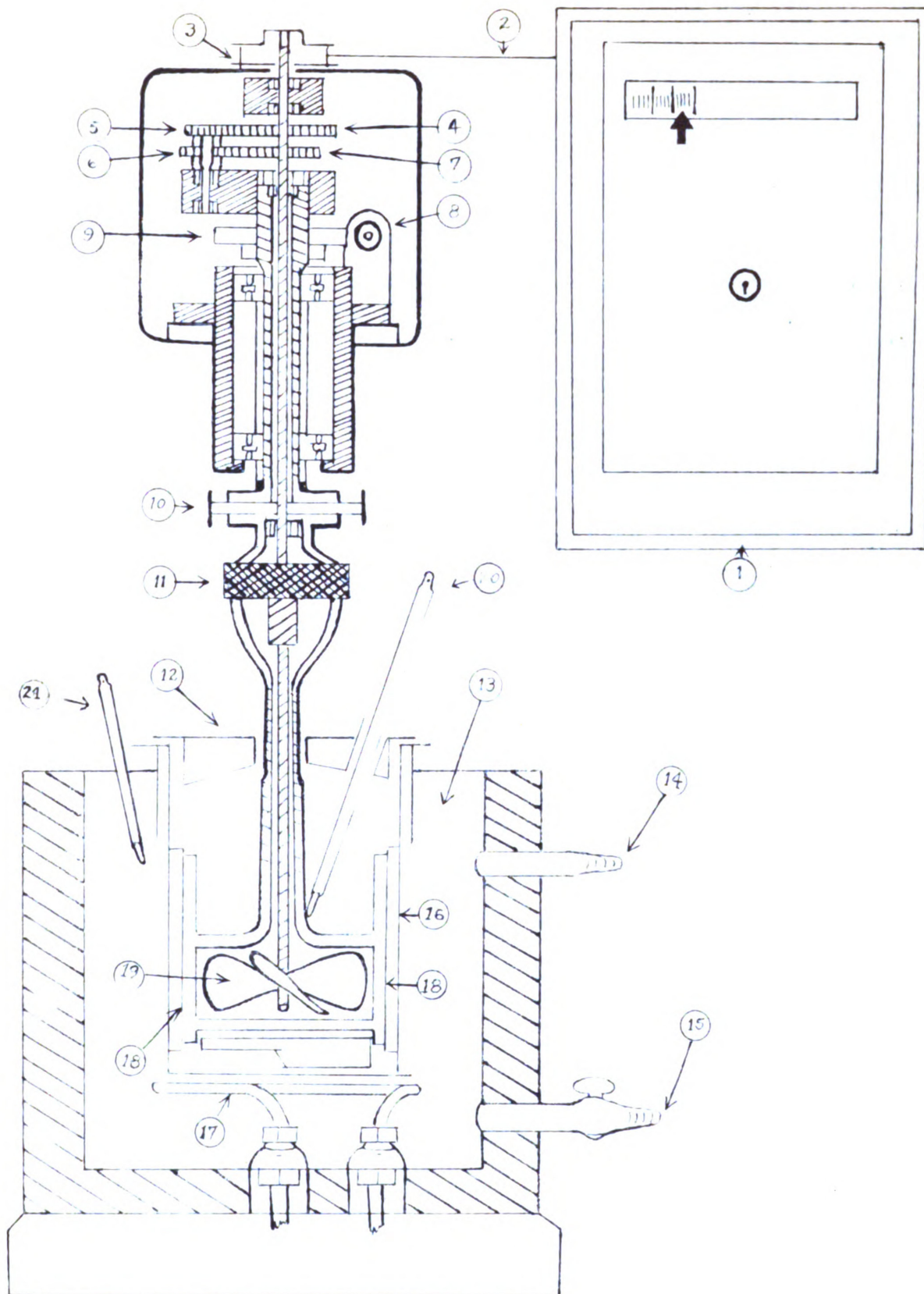


Figure 1. Sectional Drawing of the Corn Industries Viscometer.

The stirrer of the viscometer can be operated at four different speeds. However, the standard speed must be used during the gelatinization of starch pastes and is the speed recommended for viscosity measurements. This standard speed is 24 r.p.m. for the scraper and 60 r.p.m. for the propeller. The stirrer is described in the review of literature.

The basis of the viscosity determination by the Corn Industries viscometer is the measurement of the force or torque which the propeller encounters as it turns in the starch paste. This force is transmitted by a cable to the drum and, through it, to the dynamometer and recorder. A pull on the cable causes the drum to turn in a counter-clockwise direction and moves the pen across the chart from left to right. Simultaneously, the chart moves downward from the top of the recorder. The chart is marked with arcs spaced at one-half inch intervals; and, at fast speed, these arcs move at the rate of one per minute. Therefore, the chart supplies a permanent record of the viscosity changes and the length of time required for these changes to take place. Figure 2 shows a sample chart with the record of a starch paste curve. The torque can be determined mathematically (4, p. 23), or Reentel's tables (4, p. 24) can be used for transposing chart readings to torque values.

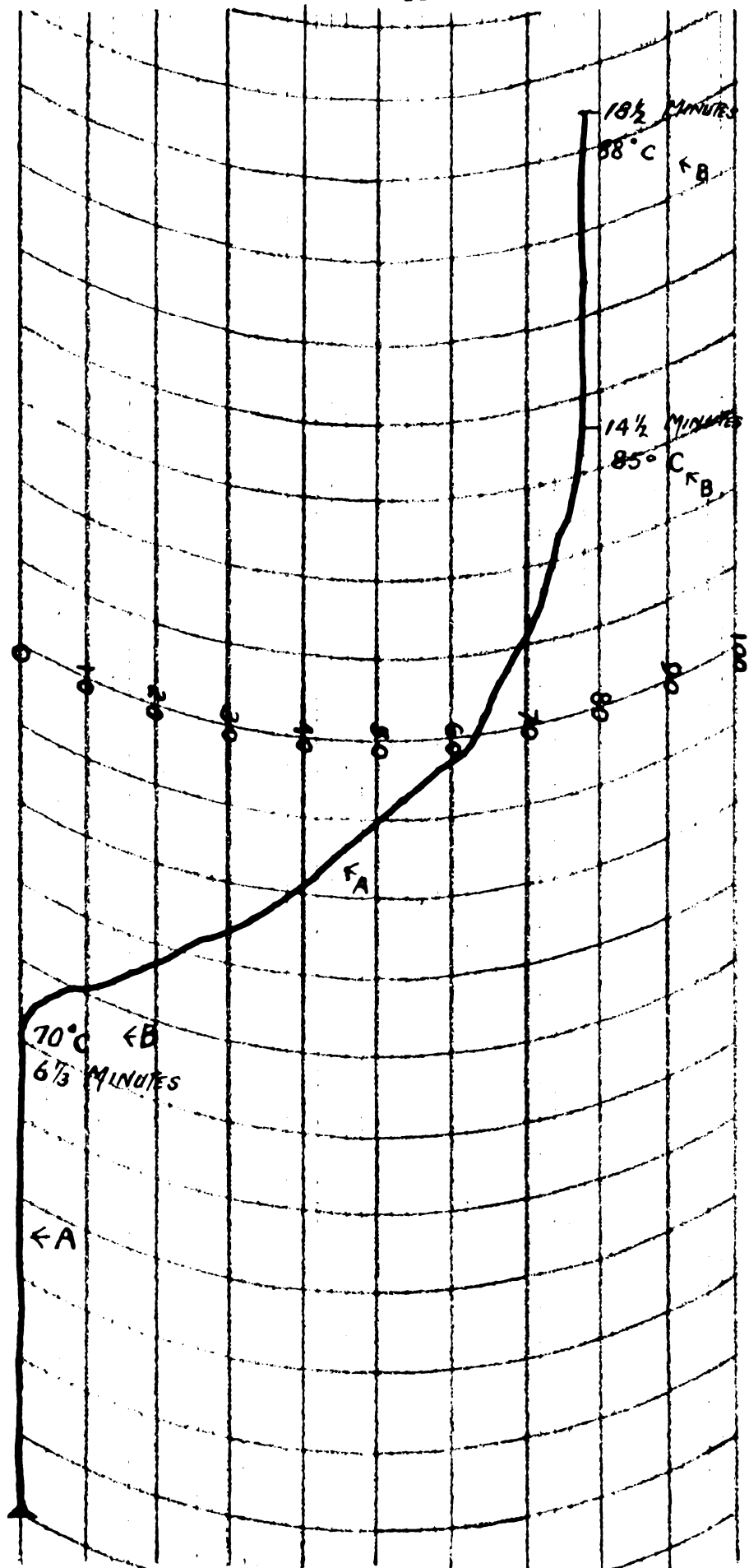


Figure 2. Sample Graph.

The weight arm of the dynamometer moves in a clockwise direction when a pull is exerted on the cable. The weight arm alone balances a torque of 275 gram-centimeters at the full scale of the chart. When highly viscous materials are cooked, the range of the viscometer can be extended by the addition of supplementary weights. These weights, at full scale, balance torques of 450, 900, and 1800 gram-centimeters. The weights can be added during a test if the material is so viscous that the recorder pen approaches the chart limit. In preliminary tests, it was found that all the pastes in this study were sufficiently viscous to warrant the use of the 1800 gram-centimeter weight. This weight was placed on the dynamometer arm and was kept there for the duration of the investigations.

The length of the cooking time of the tests depends upon the purpose of the experiments. Since the additions of varying proportions of sugar and citric acid affected the time required for the pastes to reach maximum viscosity, an identical cooking period for all the tests was found to be unsatisfactory. Because the addition of large amounts of sucrose to the starch paste caused the curve at maximum viscosity to level off very slowly, it was decided to cook all the pastes for four minutes after the peak viscosity was obtained.

A thermometer was inserted in the condenser cover in such a manner that the bulb cleared the supports of the scraper (Figure 1). This thermometer registered the temperature of the starch pastes. Observations of the paste temperature were made and recorded manually on the chart at the time of the initial viscosity rise, the maximum viscosity peak, and the end of the cooking period. (Figure 2)

Tests on cooked pastes. Since lemon pie is usually served at room temperature, it did not seem that observations on the flow of hot pastes with line spread tests would supply information of particular concern to food production managers. However, in preliminary work, line spread tests similar to those of Grawemeyer and Pfund (13) were made.

For the flow tests an 8-inch square glass plate was placed over a diagram of concentric circles made at one-half centimeter intervals. The hollow tube described by Grawemeyer and Pfund (13) was not used because the thick pastes in this study would not flow freely from such a cylinder. Instead, in the first line spread test, a Number 30 ice cream soup was filled with the hot, cooked paste and leveled with a spatula. The glass plate, at room temperature, was placed over the open end of the scoop and then both were inverted over the diagram. The paste was then released from the

scoop. This procedure was not satisfactory because the blade of the scoop was not efficient in removing all of the paste.

For a second series of flow tests, a small aluminum cake decorator tube was cut at the plunger end so that it would hold 25 cubic centimeters of the cooked paste. The tube was filled with the hot paste, leveled with a spatula, and covered with the glass plate. Plate and samples were inverted simultaneously over the diagram of concentric circles. The tube of the decorator was lifted from the plate. In all instances the paste was very thick and, in order to remove most of it from the tube, it was necessary to push the plunger down. Since this exerted pressure on the paste, the test was discarded as inaccurate.

Only in the preliminary testing was the cake decorator tube used for line spread tests on the gels after they had reached room temperature. Since there was no spread even in the softer gels, this procedure was eliminated in the final investigations.

At the end of the cooking period, the starch paste was poured into nine 3-ounce unwaxed paper cups and held at room temperature until the thermometer in one cup registered 27°C . Before the tests were made, the skin which forms on the top of cornstarch gels was removed by

cutting off one-half inch of the top of the filled paper cup with a single-edge razor blade. A sharp paring knife was then used to smooth off the cut surface of the gel sample. The gels were cut to a depth of three centimeters with the Fuchs tester (Figure 3), and the time required for cutting was recorded. Two samples were cut on the Fuchs penetrometer at 27°C, and at intervals of 1 and 2 hours after the initial cutting. For analysis of cutting data, an average of the readings on the two samples at each time interval was used.

Preliminary tests showed that a 100-gram weight was necessary to cut the starch-water and the starch-water-20 per cent sucrose gels. However, this weight traveled through the gels which were made with 40 per cent and 60 per cent sucrose with such rapidity that accurate readings were difficult to make. For this reason a 50-gram weight on the plunger was used for cutting the gels made with the higher concentrations of sucrose. These same weights were used for the gels made with these sugar concentrations for all levels of citric acid.

Figure 3. Fuchs Helistrometer

1. Plunger
2. Adjustment screw
3. Instant plunger release
4. Cutting edge of hollow cylinder
5. Level adjustment screws
6. Level eye
7. Adjustment for height of arm
8. Platform to hold weight

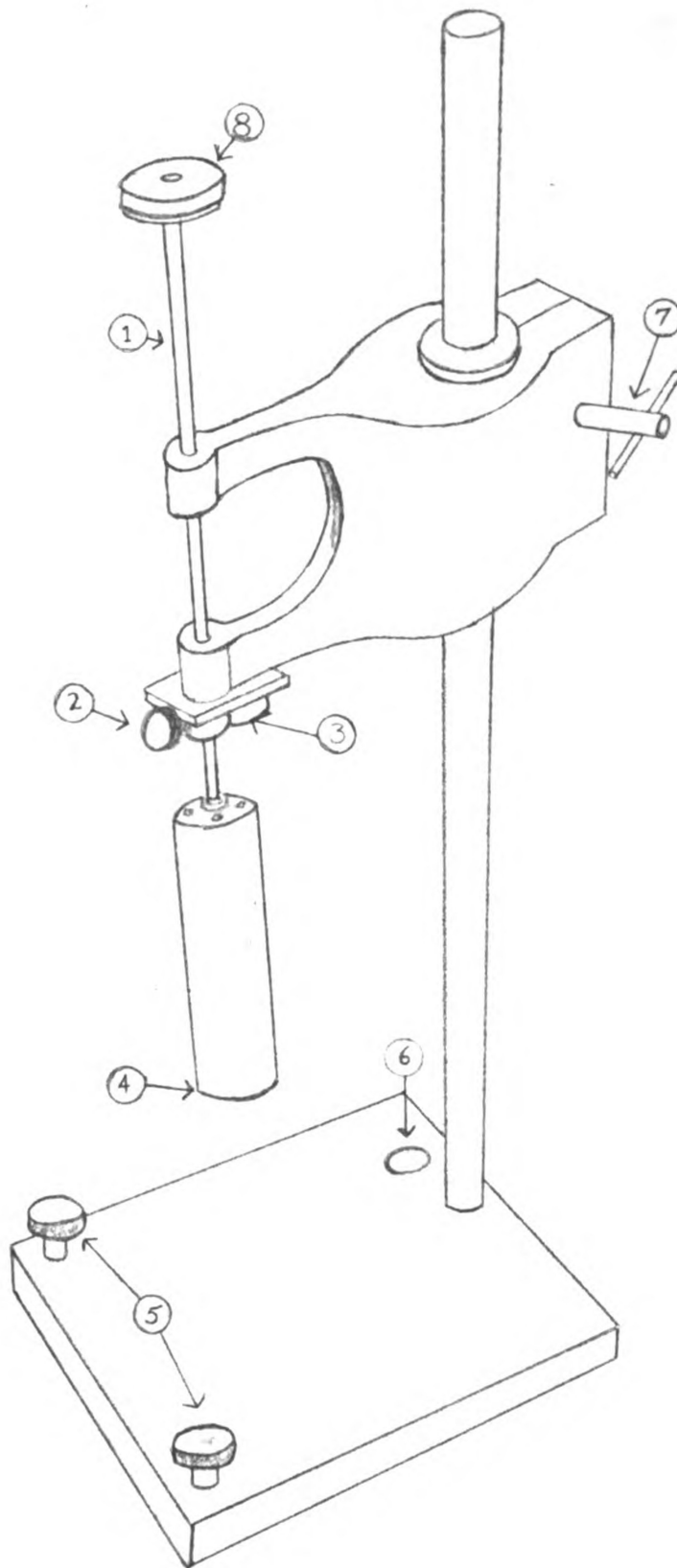


Figure 3. Fuchs Penetrometer.

DISCUSSION AND RESULTS

Viscosity Tests

Torque values. The range in torque values in the viscosity tests of the three replications of each variation are listed in Table 4. The greatest variation in torque values between

Table 4. Range in torque values in the three replications of each series.

Sugar Level %	Acid Level in Terms of Percentage of Total Liquid			
	0	40	44.5	49
0	1516 - 1530	1501 - 1516	1470 - 1501	1435 - 1501
20	1536 - 1616	1501 - 1516	1439 - 1470	1439 - 1470
40	1341	1306 - 1358*	1291 - 1324	1257 - 1324*
60	1020 - 1039	1001 - 1039	982 - 1060*	1001 - 1039

*Difference of more than 50 gram-centimeters

replications was found in the pastes made with 40 per cent and 60 per cent sucrose with acid. Where a difference of more than 50 gram-centimeters in torque values was found, the three replications were repeated. Since the same variation was found in the duplicate tests and since the time-temperature curves were identical, it appeared that the presence of large amounts of sucrose and acid could cause

a slightly greater difference in viscosity readings than those found in replicates of pastes with no sugar or with small amounts of sugar.

The averages of the torque values for the three replications in each series are listed in Table 5.

Table 5. Summary of average torque readings of the three replications in each series.

Series	Amount of 0.1 N Citric Acid in Terms of Percentage of Total Liquid	Sugar Concentration			
		0	20%	40%	60%
A	C	1521	1606	1341	1033
C	40.0	1506	1506	1335	1026
B	44.5	1491	1454	1313	1031
D	49.0	1490	1460	1291	1014

The analysis of variance of Series A, Table 6, showed that there was a highly significant difference in the

Table 6. Analysis of variance of torque readings for Series A.

Sources of Variance	D.F.	M.S.	F.
Total	11		
Between % of sucrose	3	19,293	197**
Error	8	121	

**highly significant at 1% level of probability

viscosity of the starch pastes cooked with no sucrose and those with the three concentrations of sucrose. Viscosity curves for Series A are shown in Figure 4. In this study the 40 and 60 per cent additions of sugar decreased the viscosity of the starch paste. This is in agreement with the findings of other investigators (18, 22, 40). However, it was found that the addition of 20 per cent sucrose to a 12 per cent cornstarch paste resulted in a greater viscosity than that observed in the starch and water paste. This conforms with the findings of Lester (22), who stated that 11.9 grams of sucrose cooked with 4.5 grams of cornstarch in 100 grams of water produced a greater peak in the viscosity curve than did cornstarch and water alone. She also reported that cooking the same percentage cornstarch past with 13.7, 31.6, and 35.5 grams of sucrose caused progressive decreases in the peak viscosity curves of these pastes.

The 0.1 N citric acid solution in the control series, Series E, constituted 44.5 per cent of the water volume. In Series C the citric acid was 40 per cent of the water volume, and in Series D it was 4. per cent of the water volume. The analysis of variance was made on the starch and water series without acid and with three levels of acid concentration. This analysis, Table 7, showed that

Chart
Readings

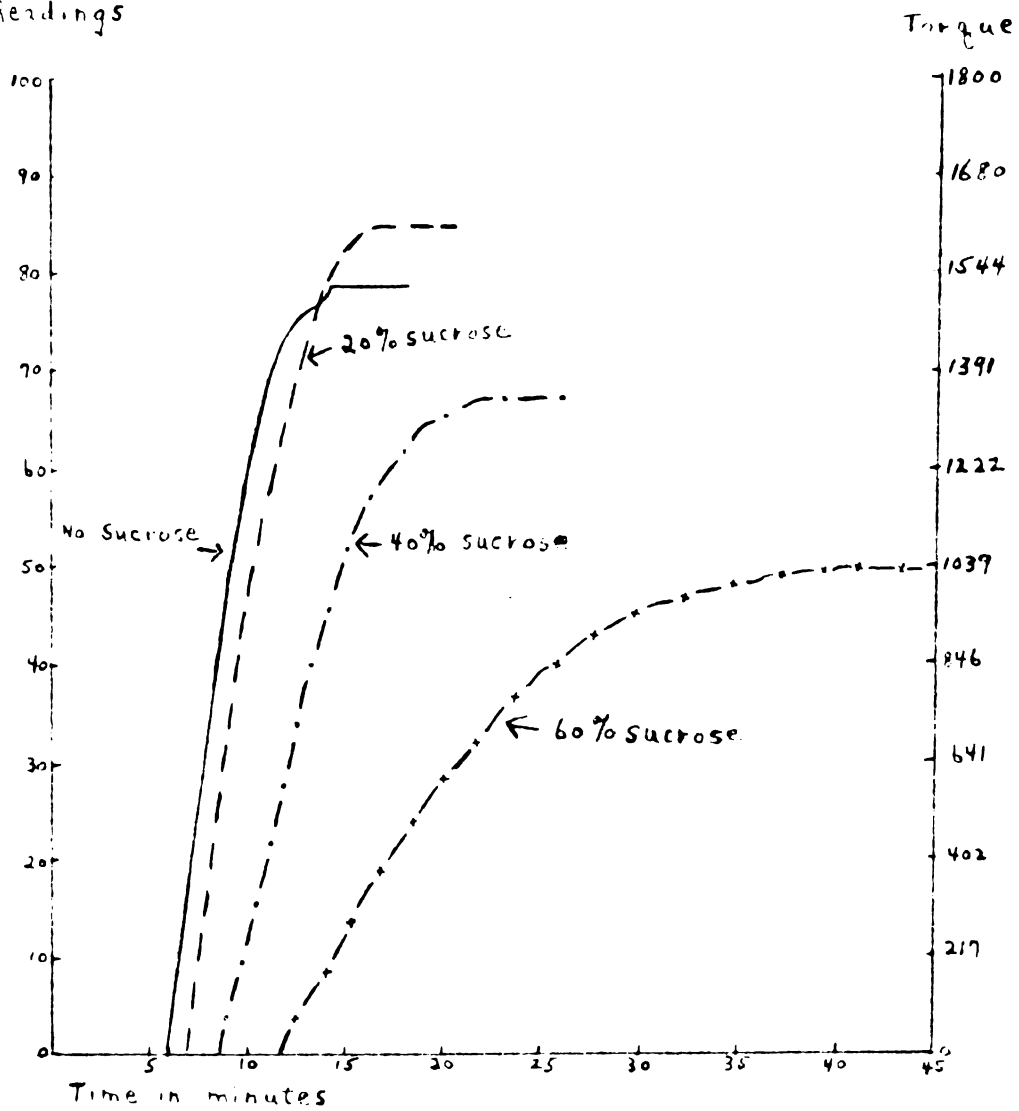


Figure 4. Effect of Sucrose Concentration on Maximum Viscosity of Cornstarch Pastes.

there was a significant difference between the treatments in this series. However, the difference in viscosity was not apparent between the starch-water and the starch-water-40 per cent acid pastes. There was no significant difference in the torque readings for viscosity of the starch-water-44.8 per cent acid and a similar paste with 40 per cent of the citric acid. The significant difference in viscosity was found only between the pastes made with no acid and the pastes made with the acid solution constituting 44.8 per cent and 40 per cent of the water volume. These pastes with high acid concentrations had a lower peak viscosity than that obtained with starch and water alone.

Table 7. Analysis of variance of torque readings for starch-water-acid pastes without sugar.

Source of Variance	D.F.	M.S.	F.
Total	11		
Between acid concentrations	3	609	4.60*
Error	8	136	

It has been mentioned that the cornstarch and water pastes made with 60 per cent sucrose had a peak viscosity

*Significant at the 1% level of probability

100-1000
60

greater than that obtained with starch and water pastes. In the 20 per cent sugar mixtures there was a highly significant progressive decrease in the peak viscosity when the pastes were cooked with citric acid solution constituting 40 per cent and 44.5 per cent of the water volume. However, there was no difference in viscosity between the pastes cooked with 41.5 per cent and 49 per cent acid concentrations. The analysis of variance for this series is shown in Table 3.

Table 3. Analysis of variance of torque readings for starch-water-acid pastes with 20% sucrose.

Source of Variance	D.F.	M.S.	F.
Total	11	.	
Between acid concentrations	3	14,815	63*
Error	8	234	

The addition of large quantities of sucrose to a cornstarch mixture has been found to cause a decrease in peak paste viscosity. In the series of viscosity tests for both 40 per cent and 60 per cent sucrose pastes, there was no significant difference between the viscosity

-----*Highly significant at the 5% level of probability

of these pastes made without acid or with any of the levels of acid concentration. The analysis of variance for the 40 per cent sucrose-acid series appears in Table 9, and for the 60 per cent sucrose-acid series in Table 10.

Table 9. Analysis of variance of torque readings for starch-water-acid pastes made with 40% sucrose.

Source of Variance	D.F.	M.S.	F.
Total	11		
Between acid concentrations	3	1581	2.8
Error	8	547	

Table 10. Analysis of variance of torque readings for starch-water-acid pastes made with 60% sucrose.

Source of Variance	D.F.	M.S.	F.
Total	11		
Between acid concentrations	3	196	0.30
Error	8	651	

The average maximum viscosity of the three replications of the starch-sucrose series are shown in Figure 5. The effect of no acid and the three levels of acid on the

average peak viscosity of the three replications of the starch-water, starch-20 per cent sucrose, starch-40 per cent sucrose, and starch-60 per cent sucrose variations is shown in Figure 6.

Temperature. The temperatures for the three replications of each variation of the pastes in all series were practically identical for initial viscosity rise, maximum viscosity peak, and four minute interval following the maximum viscosity peak. Figure 7 shows the influence of sucrose and of acid on paste temperatures at the different stages. With each increase in the proportion of sugar there was a progressive increase in the paste temperature of the initial viscosity rise, the maximum viscosity peak, and the end point of the cooking period. However, with each increase of sucrose the number of degrees of temperature rise became progressively less during the final four minutes of cooking.

The effect of citric acid on the temperature of the paste at the initial viscosity rise at the various sucrose levels was not consistent. The starch and water pastes started to thicken at 70°C when cooked without citric acid or with citric acid at each of the three levels of concentration. There was an increase from 73°C to 74°C for the temperature of initial viscosity rise in the



Figure 5. Average Maximum Viscosity of Cornstarch Pastes with Different Sucrose Concentrations. Amounts of 0.1 N Citric Acid Are Expressed in Terms of Percentage of Total Liquid Used.

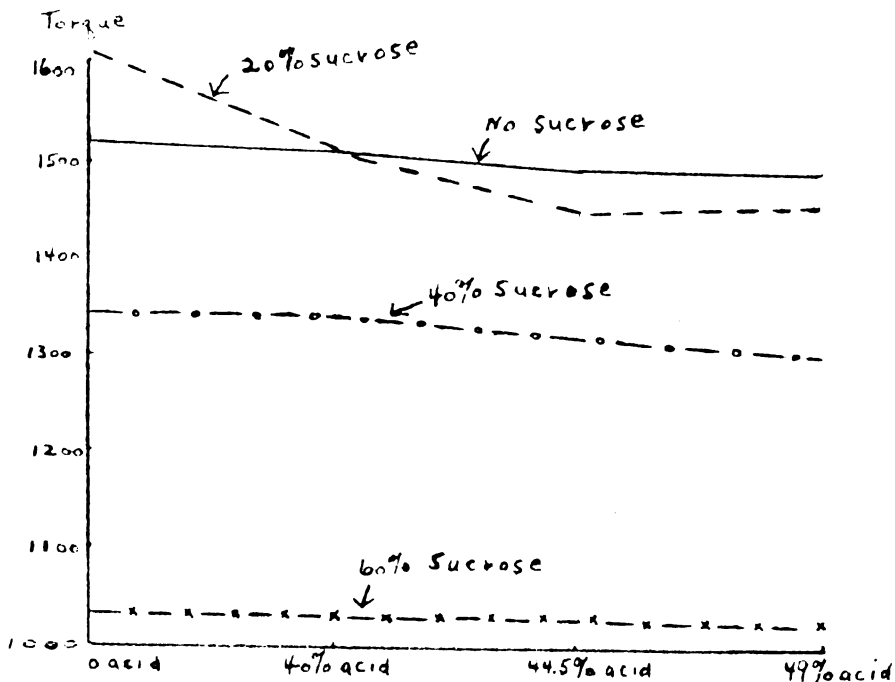


Figure 6. Average Maximum Viscosity Showing Effect of Citric Acid on Cornstarch Pastes. Amounts of 0.1 N Citric Acid Are Expressed in Terms of Percentage of Total Liquid Used.

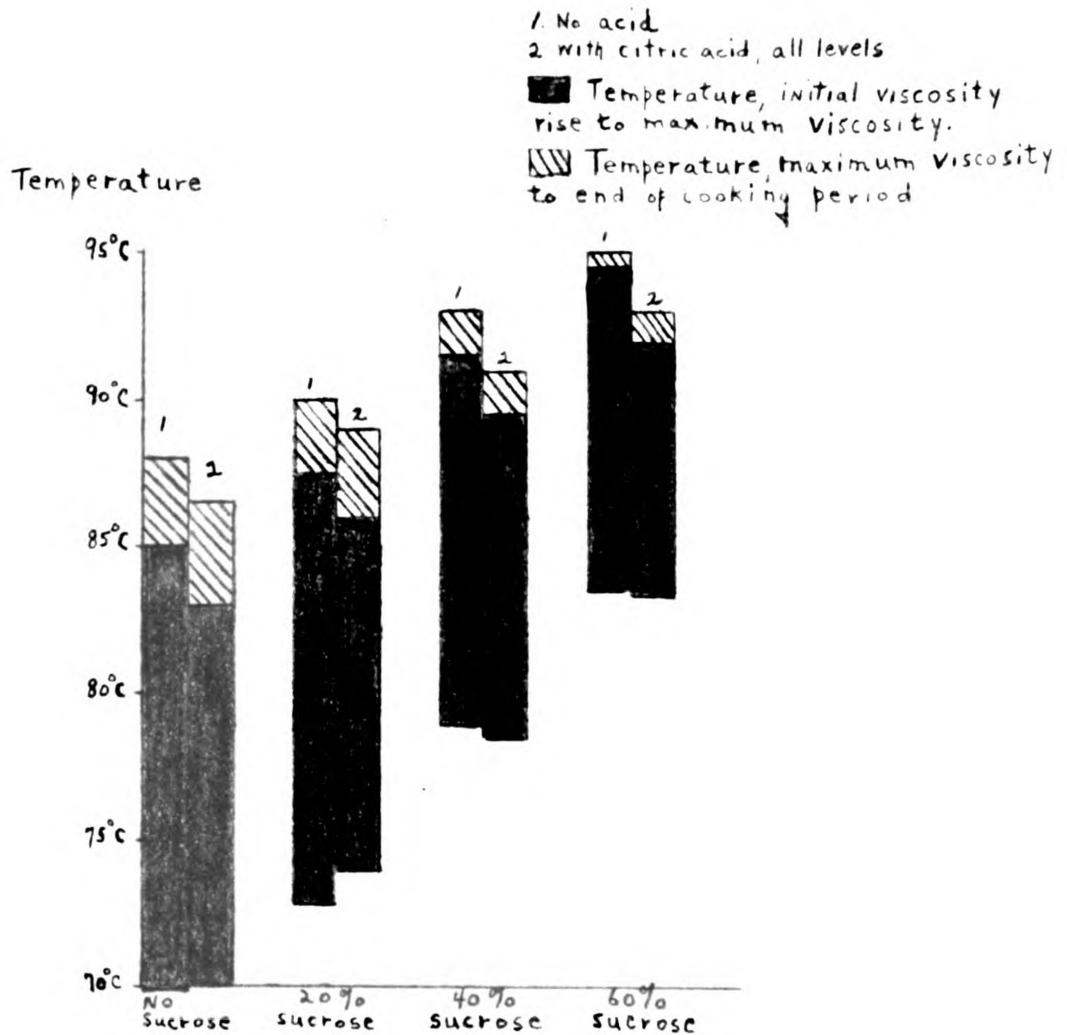


Figure 7. Effect of Sucrose Concentration and Acid on Temperature of Cornstarch Pastes.

20 per cent sucrose pastes when citric acid was added.

With the two highest sucrose concentrations, the difference in initial viscosity rise was less than one degree lower when the acid was cooked with the mixture. (Figure 7).

In all the cornstarch pastes with or without sucrose the temperature at the maximum viscosity peak and at the end of the cooking period was lower at all levels of citric acid concentration than it was in the pastes made without acid. (Figure 7)

Time. Each concentration of sucrose progressively increased the time before the initial viscosity rise. The time of this rise ranged from 6 minutes for the starch and water pastes to 11 3/4 minutes for the 60 per cent sucrose replications.

The behavior of the pastes at all levels of citric acid concentration was so similar that the effect of citric acid on the time of gelatinization may be considered the same for all the acid replications. The time of initial viscosity rise for the pastes made with and without the acid is considered practically the same. Figure 8 shows that the difference in time varied from only one-fourth to one-half minute.

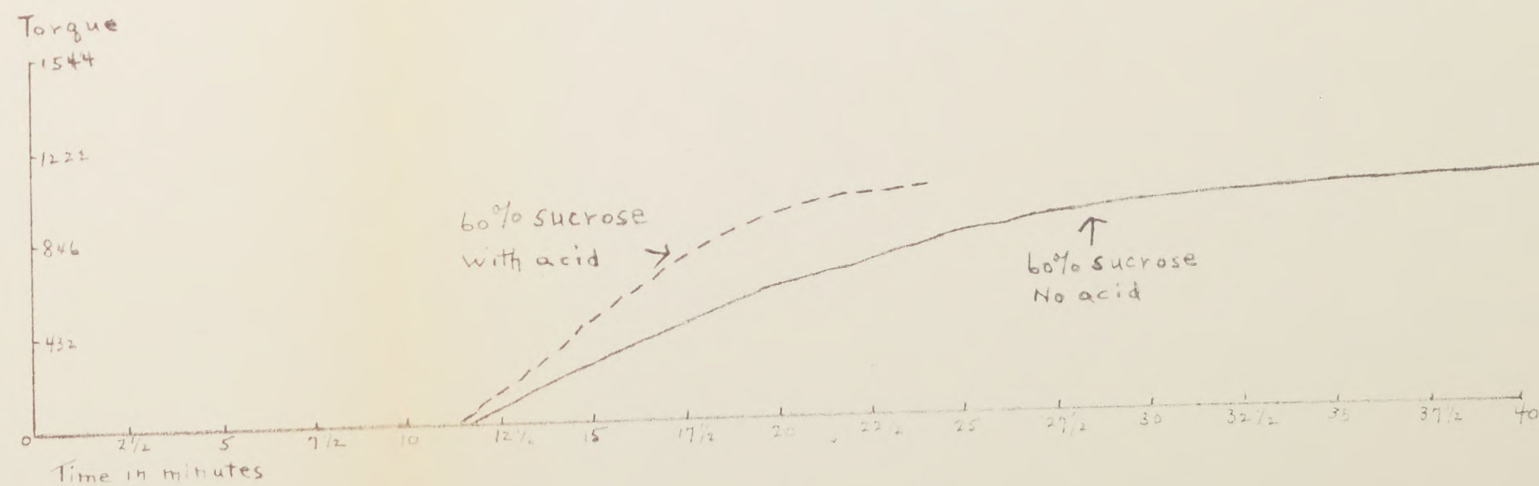
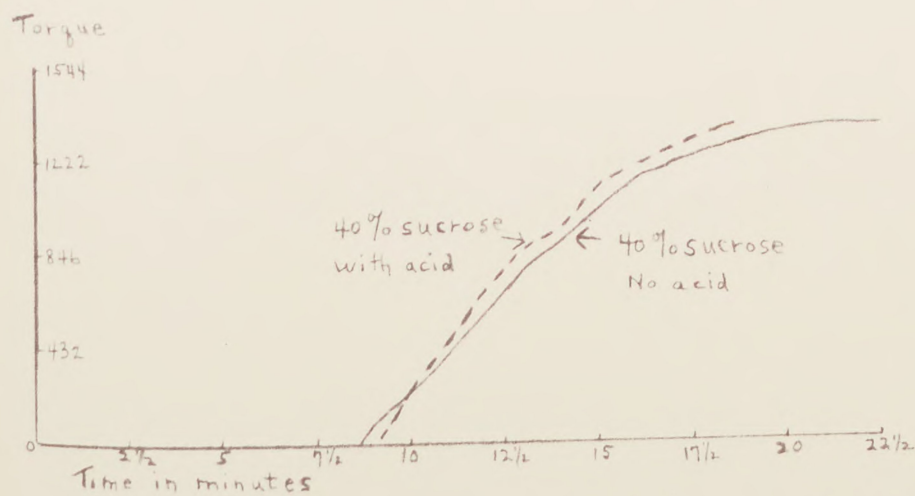
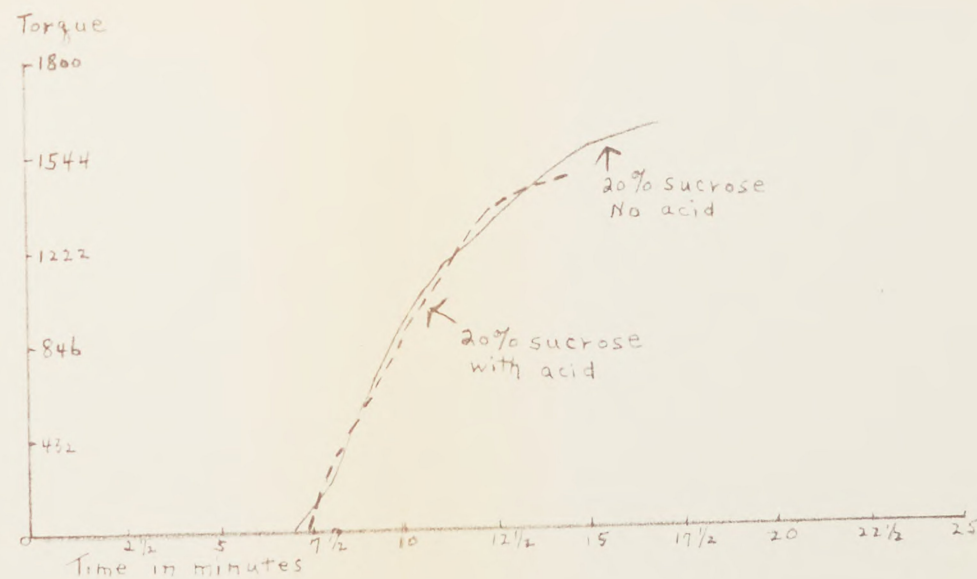
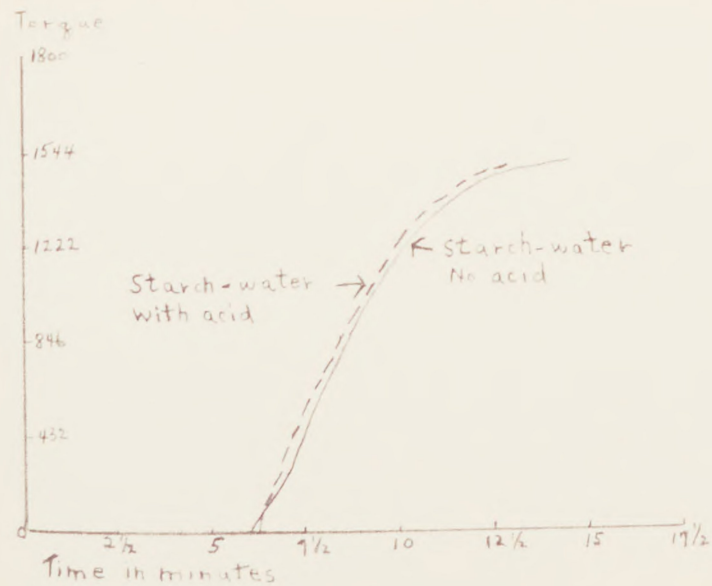


Figure 8. Effect of Sucrose Concentration and Acid on Time Required for Initial Viscosity Rise and Maximum Viscosity Point.

The time necessary to obtain maximum viscosity was greatly affected by sucrose. For the sugar pastes, Series A, the time needed to reach peak viscosity was found to be as follows:

no sucrose -----	14 3/4 minutes
20% sucrose -----	16 3/4 minutes
40% sucrose -----	22 1/2 minutes
60% sucrose -----	40 3/4 minutes

Therefore, in practical application, it is necessary to cook cornstarch pastes containing a high sugar ratio for a long time if the starch is to be gelatinized completely.

The time required for pastes at the three levels of citric acid concentration to reach maximum viscosity was quite similar. This time for reaching maximum viscosity was progressively decreased with each increment of sucrose. For the pastes which included acid, Series B, C, and D, the time needed to reach peak viscosity was averaged for the three levels of acid. These averages are as follows:

no sucrose -----	13 minutes
20% sucrose -----	14 1/2 minutes
40% sucrose -----	18 1/2 minutes
60% sucrose -----	24 minutes

Figure 8 shows the time required to obtain maximum viscosity in the pastes in Series A, B, C, and D.

Gel Strength Tests

Effect of time of holding after cooling. The samples of cornstarch paste used for gel strength tests were held at room temperature. The first cutting test, with the Pouns penetrometer, was made at the time the pastes reached 27°C. Similar cutting tests were made one and two hours later. Two samples were cut at each time interval and the cutting time, in seconds, was averaged for the two readings. The analysis of variance was made using the two sources of variance: (1) the cutting time at each time interval and, (2) the cutting time of the three replications.

The averages in cutting time of the three replications at each time interval for Series A are listed in Table 11. This table also listed the F values of the analysis of variance in which the sources of variance were the average cutting time of each replication and the cutting time at each time interval.

The analysis of variance showed that the gel strength of the starch-and-water pastes were the same at 27°C and at intervals of one hour and two hours later. There was a significant difference attributable to time intervals in the gel strength of the pastes made with 20 per cent sucrose. The cutting time at the end of one hour increased greatly

over that at a temperature of 27°C. However, average cutting time did not increase appreciably after the one

Table 11. Average cutting time and F values of analysis of variance in the starch-water-sucrose series. No sugar and 20% sucrose pastes cut with 100-gram weight; 40% and 60% sucrose pastes cut with 200-gram weight.

Sugar Concen- tration	Average cutting time of three replications of Series A (in seconds)			F value Sources of variance, time intervals vs. replications
	Cutting Intervals			
	27°C 1 hr. later		2 hr. later	
No sugar	23	23	21	0.19
20% sugar	6	11	11	0.14*
40% sugar	28	42	61	85.80**
60% sugar	11	11	16	70.00**

hour interval. The cornstarch pastes made with 40 per cent and 60 per cent sucrose showed a highly significant difference in gel strength attributable to time. The cutting time was progressively longer for these pastes at both the one hour and the two hour period after the temperature of the pastes had reached 27°C.

*Significant at 5% level of probability
**Highly significant at 1% level of probability.

Similar cutting tests were made on the replications of the starch-water and the starch-water-sucrose pastes at all levels of citric acid concentration. The average cutting time, in seconds, for the three replications of these pastes at each time interval are listed in Table 12. This table also contains the F value of the analysis of variance in which the sources of variance were the cutting time of each replication and the cutting time at each time interval. The analyses of variance showed that the gel strength was significantly greater at one and two hours after the pastes had reached 27°C for the following four variations:

- 1) no sugar, 40 per cent citric acid
- 2) 20 per cent sucrose, 40 per cent acid
- 3) 20 per cent sucrose, 40 per cent acid
- 4) 40 per cent sucrose, 40 per cent acid

There was a highly significant increase in the gel strength of 40 per cent sucrose pastes made without acid and with the two height levels of acid concentration between the cutting intervals. The cutting time was also significantly greater at each time interval for the 60 per cent sucrose pastes made with 40 per cent and 45 per cent citric acid. Although the figures in Table 12 show that there was an increase in cutting time at each time interval for the pastes made with 60 per cent sucrose and 44.5 per cent

citric acid, this difference was not significant because there was greater variation in the cutting time between the replications of these pastes. For this same reason

Table 18. Average cutting time in seconds of three replications at three time intervals of starch-water and starch-water-sucrose pastes at three levels of citric acid concentration. No sucrose and 5.5% sucrose pastes cut with 150-gram weight; 40% and 60% sucrose pastes cut with 80-gram weight.

Average cutting time for three replications of starch-water and starch-water-sucrose pastes at all levels of acid concentration (in seconds)					F value Sources of variation, time intervals vs. replications
A sugar	B acid ¹	17°C	1 hr. later	2 hr. later	
0	0	23	43	41	0.19
0	40	11	15	15	4.67
0	44.5	15	20	19	4.00
0	49	11	13	13	9.10*
20	0	6	11	11	6.14*
20	40	7	5	15	12.10*
20	44.5	15	12	13	2.00
20	49	11	11	14	7.00*
40	0	22	42	61	69.00**
40	40	26	111	132	9.45*
40	44.5	73	133	123	10.00**
40	49	42	133	131	20.25**
60	0	10	11	13	70.00**
60	40	7	20	24	49.00**
60	44.5	11	17	20	4.40
60	49	10	16	21	35.09**

*Significant at 5% level of probability

**Highly significant at 1% level of probability

¹Acid Level in Terms of Percentage of Total Liquid

the analysis showed no difference in the strength of the gel at the time it reached 27°C , 1 hour and 2 hours later for the starch-water pastes without acid and with 40 per cent acid and for the starch-water-20 per cent sucrose paste made with 44.5 per cent acid. Figure 9 shows the effect of aging on the gel strength of unsweetened and sweetened cornstarch paste with varying acid levels.

Effect of sucrose. It was necessary to use a 100-gram weight on the plunger of the Fuoss penetrometer to cut the starch-water and the starch-water-20 per cent sucrose pastes. Since this weight traveled through the gels which were made with 40 per cent and 60 per cent sucrose with such rapidity that accurate readings could not be made, a 50-gram weight instead of a 100-gram weight was used to cut these gels.

An analysis of variance was made on the cutting time of the pastes made with starch-water and starch-water-20 per cent sucrose. A similar analysis was made on the cutting time of these two variations at each level of acid concentration. These analyses appear in the appendix, Tables 14 through 17. There was a highly significant difference in the gel strength between the starch-water and starch-water-20 per cent sucrose pastes when these

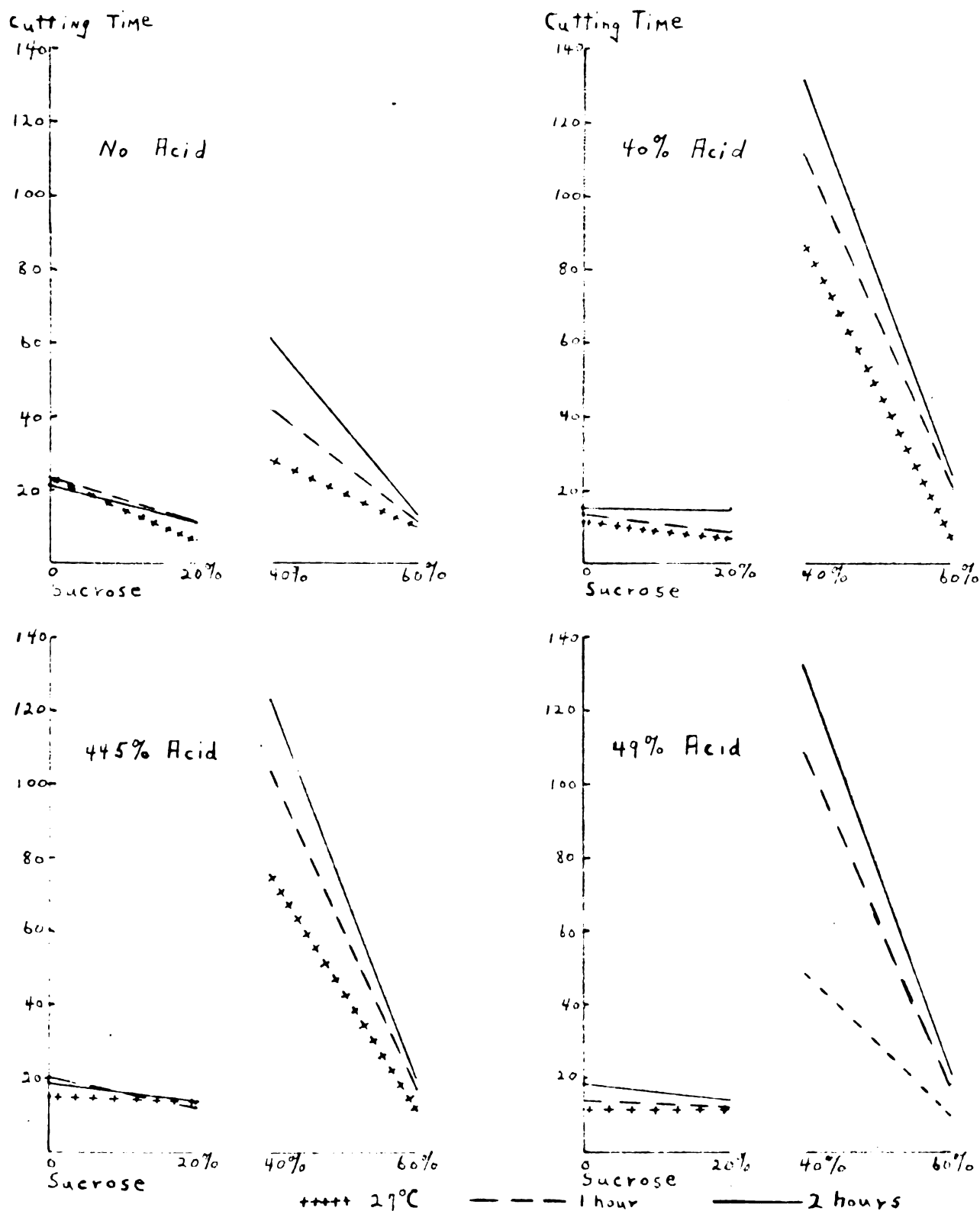


Figure 9. Effect of Time on Gel Strength with Varying Acid Levels. 0 and 20% Sucrose Cut with 100-Gm. Weight; 40 and 60% Sucrose Cut with 20-Gm. Weight. Amounts of 0.1 N Citric Acid Are Expressed in Terms of Percentage of Total Liquid Used.

pastes were made without acid and with 44.6 per cent acid, and a significant difference when they were made with 40 per cent acid. However, there was no difference in the gel strength of the cornstarch pastes without sugar and with 60 per cent sugar when the acid concentration was 40 per cent of the water volume.

The analyses of variance for the comparison of the cutting time between the gels made with starch-water-40 per cent sucrose and with starch-water-60 per cent sucrose without acid and at the three levels of acid concentration are listed in the appendix, Tables 1 through 31. There was a highly significant difference in gel strength of the pastes made with 40 per cent and those made with 60 per cent sucrose, without acid and at all levels of acid concentration. The pastes made with the 60% sucrose

Table 1. Average cutting time of three replications of cornstarch gels (in seconds).

Sugar	Acid Level in Terms of Percentage of Total Liquid											
	40%			49%			44.6%			48%		
	Time			Time			Time			Time		
	27°	1 hr	2 hr	27°	1 hr	2 hr	27°	1 hr	2 hr	27°	1 hr	2 hr
0	23	43	71	11	13	15	15	19	11	13	13	16
20%	6	11	11	7	9	15	13	13	11	11	11	14
40%	28	43	61	36	111	133	75	103	133	49	103	131
60%	13	11	13	7	15	14	11	17	15	10	13	21

concentration, without acid and at all levels of acid concentration formed a weaker gel than pastes made with 40 per cent sucrose. This is evident in the great decrease in the time needed to cut the gels which were made with 60 per cent sucrose, Table 16, compared with that required to cut the gels containing 40 per cent sucrose.

The effect of sugar concentrations on gel strength at varying concentrations of acid is shown in Figure 11.

Effect of acid. An analysis of variance was made on the cutting time for the starch-water gels without acid and at all levels of acid concentration. (Appendix, Table 82) The sources of variance were the replications, the treatments with and without acid, and the cutting time at each time interval. The analysis includes the interaction between the effect of time and the effect of acid.

Similar analysis of variance were made on the sweetened cornstarch pastes without acid and at the three levels of acid concentration (Appendix, Tables 83 through 85).

There was a highly significant difference in the gel strength of the starch-water pastes made without acid and those made with the citric acid solution at the three levels of acid concentration. The average cutting time for the starch-water pastes was significantly greater than the cutting time of similar pastes made with acid.

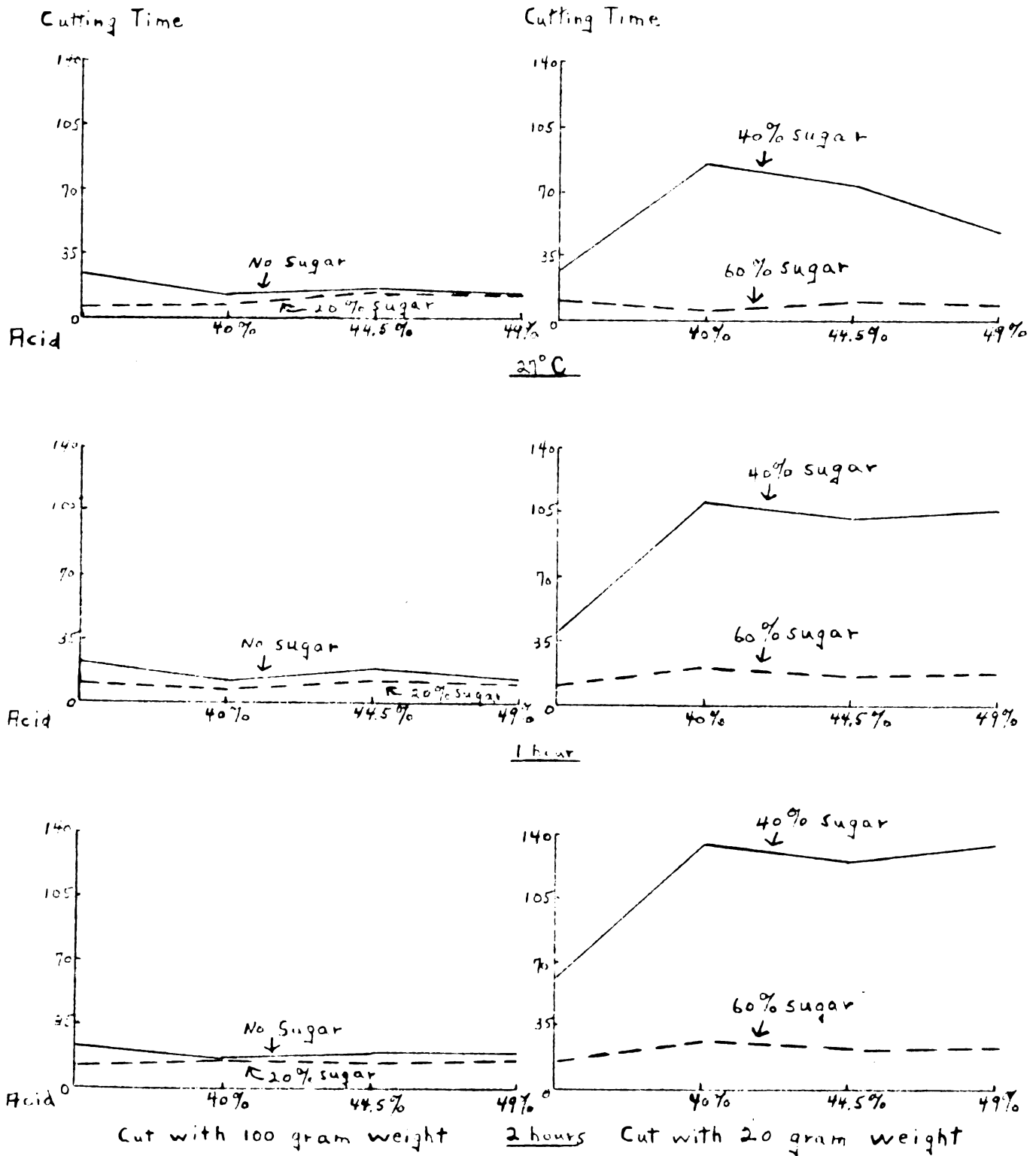


Figure 10. Effect of Sugar Concentration on Gel Strength at Varying Concentrations of Acid. Amounts of 0.1N Citric Acid Are Expressed in Terms of Percentage of Total Liquid Used.

It appears from the analysis of the cutting tests that the citric acid was the contributing factor in the decrease in gel strength of the cornstarch-water pastes.

All sweetened starch pastes made with all concentrations of citric acid solution showed a highly significant increase in gel strength over similar pastes made without acid. There is also a highly significant increase in gel strength of these sweetened pastes made with citric acid attributable not only to acid but also to the effect of time interval. Therefore, both the addition of acid and the length of time the pastes were held before cutting, contributed to the increase in gel strength of all the cornstarch pastes made with sucrose.

The effect of the concentration of citric acid on gel strength at the different time intervals is shown in Figure 11.

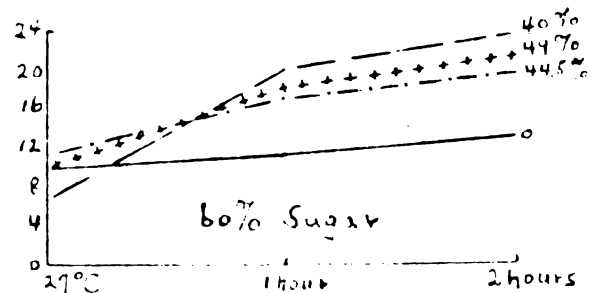
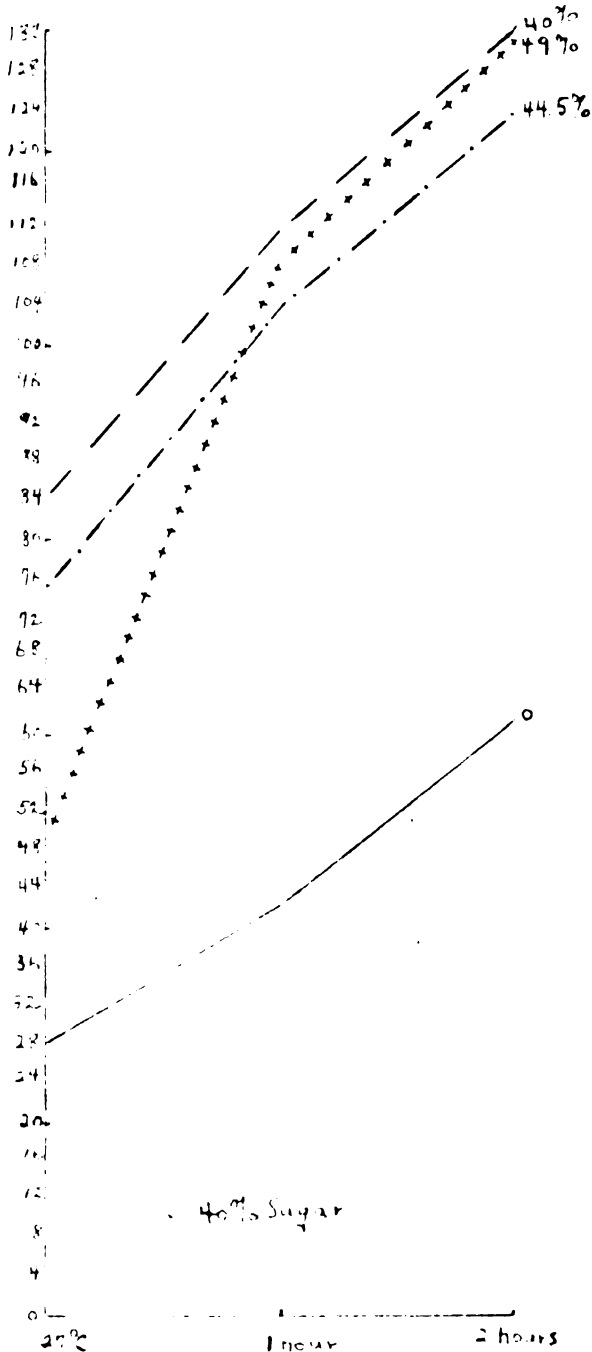
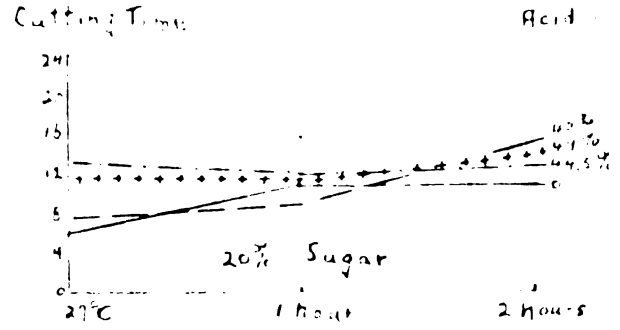
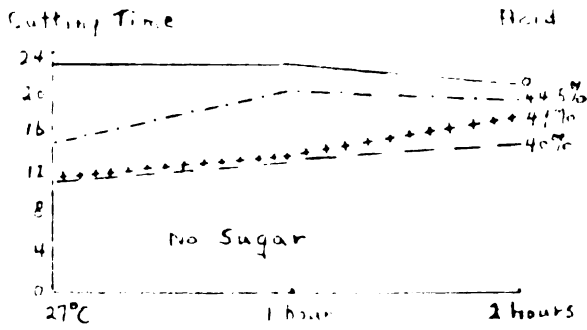


Figure 11. Effect of Concentration of Acid on Gel Strength at Different Time Intervals. Zero and 20% Sucrose Cut with 100-Gram Weight; 40 and 60% Sucrose Cut with 20-Gram Weight. Amounts of 0.1 N Citric Acid Are Expressed in Terms of Percentage of Total Liquid Used.

SUMMARY

The maximum viscosity of a 20 per cent cornstarch paste increased greatly with the addition of 40 per cent sucrose, and progressively decreased by the addition of 40 per cent and 60 per cent sucrose.

No significant difference in maximum viscosity was found between cornstarch-water pastes without acid and with 40 per cent 0.1 N citric acid solution. Additions of 44.3 per cent and 49 per cent citric acid lowered the maximum viscosity.

The additions of 40 per cent and 44.3 per cent citric acid solution appreciably lowered the viscosity of the 40 per cent sucrose-cornstarch pastes, but the addition of 49 per cent acid did not further lower the maximum viscosity. There was no apparent difference in the maximum viscosity of the 40 per cent sucrose pastes attributable to acid, nor was any such difference noted in the 60 per cent sucrose pastes.

With each increase in the proportion of sugar there was a progressive increase in the temperature of the paste at the initial viscosity rise, maximum viscosity peak, and end point of cooking period. The addition of citric acid at all levels did not affect appreciably the temperature of the initial viscosity rise of either the unsweetened

(2)

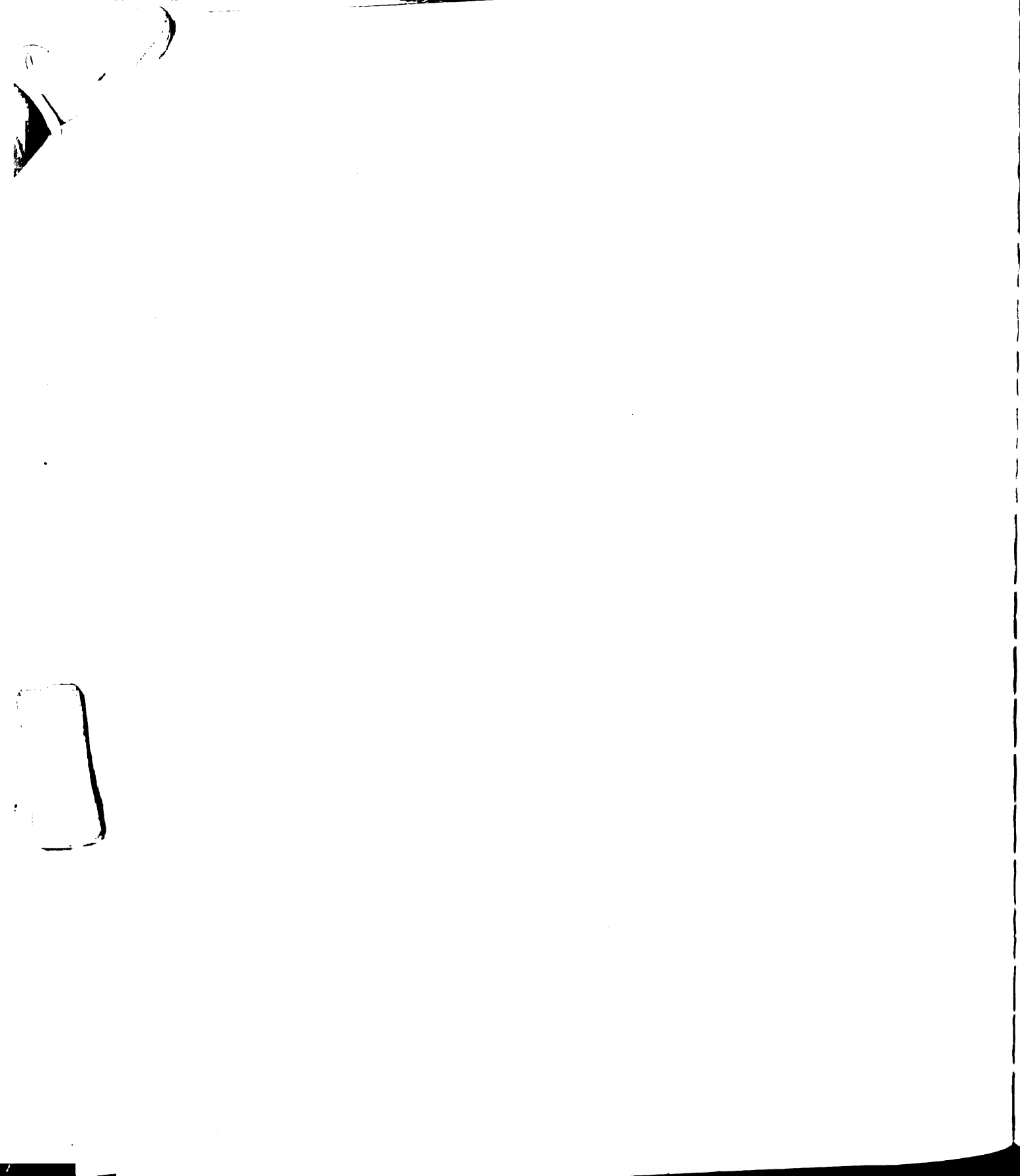


or the sweetened starch pastes. However, in all the pastes, the temperature at the maximum viscosity peak and the end point of the cooking period was lower at all levels of acid concentration than it was in the pastes made without acid.

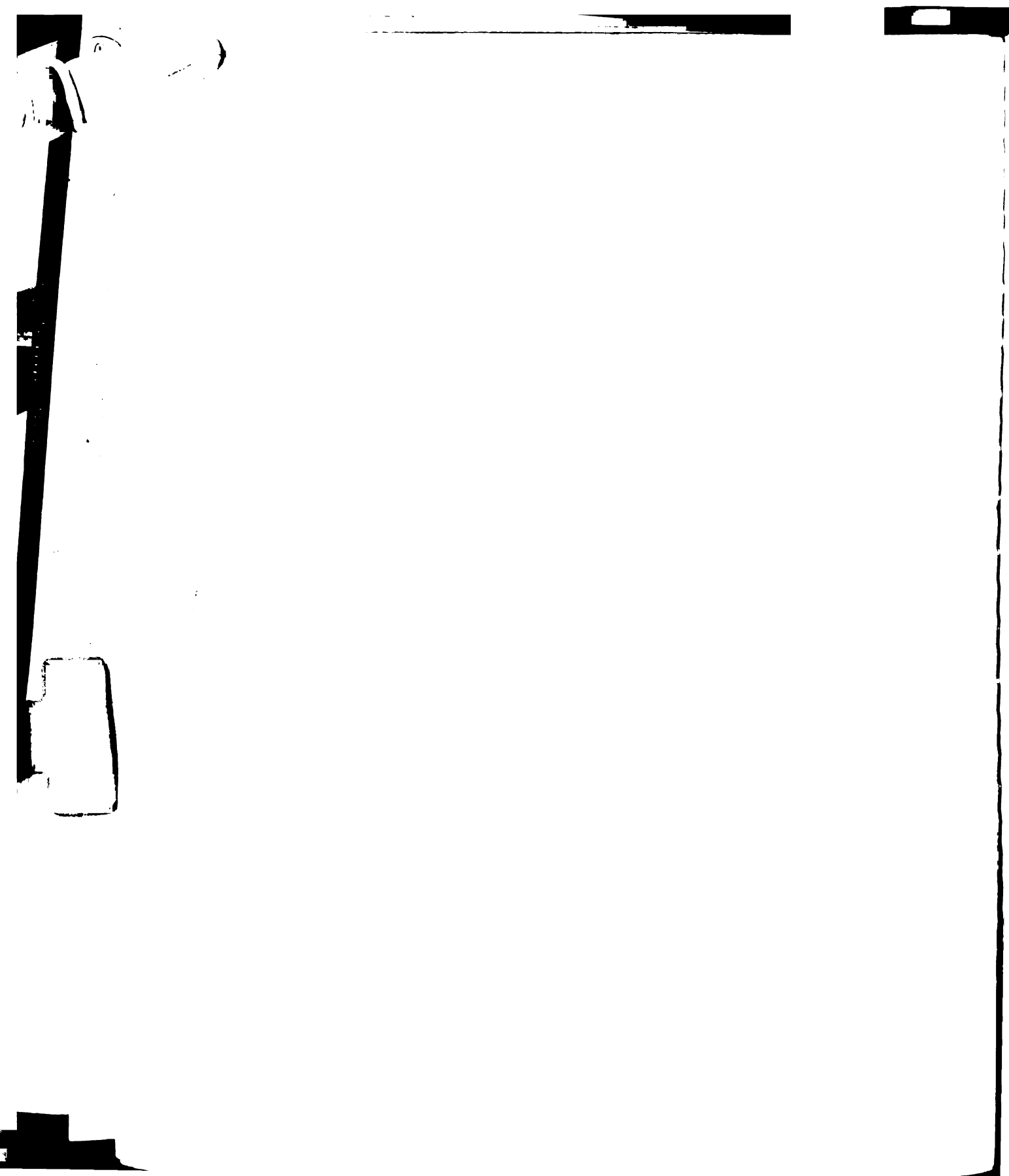
When concentration of sucrose progressively increased the time required for initial viscosity rise. The addition of citric acid to the unsweetened and the sweetened pastes did not affect the time of the initial viscosity rise.

Each increment of sucrose progressively increased the time required to obtain maximum viscosity; the time varied from 14 3/4 minutes for the starch-water mixtures to 43 3/4 minutes for the starch-water-60 per cent sucrose pastes. At all levels of citric acid concentration the time necessary to obtain maximum viscosity progressively decreased as the concentration of sugar increased. The time required to reach peak viscosity with the citric acid ranged from 13 minutes for the starch-water pastes to 14 minutes for the 60 per cent sucrose pastes.

The gel strength of neither the starch-water pastes nor the starch-water-acid pastes appeared to be affected by aging of the pastes. However, there was a progressive increase in gel strength on standing for the pastes at all levels of sugar concentration. In general, the sweetened pastes, made with the addition of acid also showed an increase in gel strength attributable to time interval.



There was a progressive decrease in gel strength of the pastes with each increment of sugar. The gel strength of starch-water-acid pastes was significantly less than that of starch-water pastes. However, there was an appreciable increase in gel strength of all the sweetened pastes made with the addition of citric acid, with an additional increase in strength attributable to aging of the pastes.



CONCLUSIONS

The amount of cornstarch needed for a specific viscosity cannot be arbitrarily set by determining the percentage of thickening agent to the volume of water without taking into consideration the percentage of other ingredients. In this limited study it was found that when sucrose constituted 20 per cent of the weight of the liquid, a quantity approximating the amount of sugar in many thickened and sweetened desserts, the viscosity of the mixture increased. However, extremely large proportions of sugar decreased the viscosity of a cornstarch paste made with or without acid.

A higher temperature and a much longer cooking period are required to obtain maximum viscosity of cornstarch pastes when increased proportions of sugar are used. To obtain complete gelatinization of the cornstarch, pastes made with a high proportion of sugar require a longer cooking time than pastes with low sugar content.

All levels of citric acid decreased the viscosity of pastes made with no sugar or with 20 per cent sugar. However, in this limited study, acid did not decrease the viscosity of cornstarch pastes made with 40 and 60 per cent sugar. Since recipes for lemon pie fillings generally contain sugar within the 40 to 60 per cent range and acid

substances at a level of 40 percent or lower, it seems feasible to consider carefully the reasons commonly given for the procedures recommended for such fillings. On the basis of the findings of this study, it appears that the accepted theory, based on the premise that the hydrolyzing effect of starch prevents the thickening of sweetened starch pastes, may not be as well substantiated as heretofore believed.

The gel strength of all the sweetened cornstarch pastes increased when these pastes were cooked with citric acid. There is a widely prevalent belief that sugar-starch pastes cooked with acid yield softer gels than sugar-starch pastes cooked without acid. However, the results of this study indicated that the addition of acid at the beginning of the cooking period gave pastes with greater gel strength than similar pastes cooked without acid.

Total cooking time is frequently an important factor in preparation of sweetened pastes in quantity food production. From this limited study it appears that the addition of acid substances at the beginning of the cooking of such pastes makes it possible to obtain maximum viscosity in an appreciably shorter time than is required for sugar-starch pastes alone.

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APPENDIX

Table 11. Analysis of variance of the cutting time at each time interval of the three replications of starch-water compared with starch-water-20% sucrose.

Source of variation	D.F.	M.S.	F value
Total	17		
Replications	4		
Time intervals	3	9.9	
Time { starch-water vs. { starch-water-20% sucrose	2	17.0	
Treatment { starch-water vs. { starch-water-20% sucrose	1	721.0	45.10**
Error	8	15.0	

Table 12. Analysis of variance of the cutting time at each time interval of the three replications of starch-water-10% citric acid compared with starch-water-20% sucrose-40% citric acid.

Source of variation	D.F.	M.S.	F value
Total	17		
Replications	4		
Time intervals	3	32	
Time { Starch-water-40% acid { vs. starch-water-20% { sucrose-40% acid	2	7	
Treatments { Starch-water-40% acid { vs. starch-water-20% { sucrose-40% acid	1	30	10*
Error	8	3	

*Highly significant at 1% level of probability

**Significant at 5% level of probability

Table 16. Analysis of variance of the cutting time at each time interval of the three replications of starch-water-44.5% citric acid compared with starch-water-20% sucrose-44.5% citric acid.

Source of variation	D.F.	M.S.	F value
Total	17		
Replications	4		
Time intervals	2	10.00	
Time { starch-water-44.5% acid vs. starch-water-20% sucrose- 44.5% acid	2	14.00	
Treatment { starch-water-44.5% acid vs. starch-water-20% sucrose-44.5% acid	1	130.00	48.2**
Error	3	2.00	

Table 17. Analysis of variance of the cutting time at each time interval of the three replications of starch-water-4% citric acid compared with starch-water-20% sucrose-4% citric acid.

Source of variation	D.F.	M.S.	F value
Total	17		
Replications	4		
Time intervals	2	40.00	
Time { starch-water-4% acid vs. starch-water-20% sucrose-4% acid	2	3.30	
Treatment { starch-water-4% acid vs. starch-water-20% sucrose-4% acid	1	14.00	4.7
Error	3	2.80	

**highly significant at 1% level of probability

1

Table 11. Analysis of variance of the cutting time at each time interval of the three replications of starch-water-40% sucrose compared with starch-water-60% sucrose.

Source of variation	D.F.	M.S.	F value
Total	17		
Replications	4		
Time intervals	2	489.16	
Time { Starch-water-40% sucrose vs. starch-water-60% sucrose	2	304.47	
Treatment { Starch-water-40% sucrose vs. starch-water-60% sucrose	1	4809.10	9.83**
Error	3	4.6	

Table 12. Analysis of variance of the cutting time at each time interval of the three replications of starch-water-40% sucrose-40% citric acid compared with starch-water-60% sucrose-40% citric acid.

Source of variation	D.F.	M.S.	F value
Total	17		
Replications	4		
Time intervals	2	1492.50	
Time { Starch-water-40% sucrose- 40% acid vs. starch-water- 60% sucrose-40% acid	2	313.00	
Treatment { Starch-water-40% sucrose- 40% acid vs. starch-water- 60% sucrose-40% acid	1	4049.00	419.9**
Error	3	89.83	

*Highly significant at 1% level of probability

Table 20. Analysis of variance of the cutting time at each time interval of the three replications of starch-water-40% sucrose-44.5% citric acid compared with starch-water-60% sucrose-44.5% citric acid.

Source of variation	D.F.	M.S.	F value
Total	17		
Replications	4		
Time intervals	2	1930.5	
Time { starch-water-40% sucrose- 44.5% acid vs. starch- water-60% sucrose-44.5% acid	2	557.0	
Treatment { starch-water-40% sucrose- 44.5% acid vs. starch- water-60% sucrose-44.5% acid	1	32,004.0	575.65**
Error	3	56.5	

Table 21. Analysis of variance of the cutting time at each time interval of the three replications of starch-water-40% sucrose-49% citric acid compared with starch-water-60% sucrose-49% citric acid.

Source of variation	D.F.	M.S.	F value
Total	17		
Replications	4		
Time intervals	2	3332.0	
Time { starch-water-40% sucrose- 49% acid vs. starch-water- 60% sucrose-49% acid	2	2017.5	
Treatment { starch-water-40% sucrose- 49% acid vs. starch- water-60% sucrose-49% acid	1	29040.0	286.11**
Error	3	101.5	

**highly significant at 1% level of probability

Table 22. Analysis of variance starch-water at all levels of citric acid concentration. Interaction of effect of acid and cutting time at each time interval.

Source of variation	D.F.	M.S.	F value
Total	35		
Replications	8	63.53	0.58**
Acid	3	160.67	16.63**
Time intervals	2	30.00	3.11
Acid vs. time	6	14.72	1.52
Error	16	9.66	

Table 23. Analysis of variance starch-water-20% sucrose at all levels of citric acid concentration. Interaction of effect of acid and cutting time at each time interval.

Source of variation	D.F.	M.S.	F value
Total	35		
Replications	8	4.66	2.09
Acid	3	18.33	8.30**
Time intervals	2	51.50	23.09**
Acid vs. time	6	10.83	4.80**
Error	10	2.23	

**highly significant at 1% level of probability

12

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12

Table 24. Analysis of variance starch-water-40% sucrose at all levels of citric acid concentration. Interaction of effect of acid and cutting time at each time interval.

Source of variation	D.F.	M.S.	F value
Total	35		
Replications	8	431.86	3.87*
Acid	3	6920.33	62.02**
Time	2	6705.00	60.09**
Acid vs. time	6	900.77	8.07**
Error	16	111.59	

Table 25. Analysis of variance starch-water-60% sucrose at all levels of acid concentration. Interaction of effect of acid and cutting time at each time interval.

Source of variation	D.F.	M.S.	F value
Total	35		
Replications	8	30.53	5.17**
Acid	3	61.80	10.47**
Time	2	317.20	53.76**
Acid vs. time	6	27.75	4.70**
Error	16	5.90	

*Significant at 5% level of probability

**Highly significant at 1% level of probability.

3



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