1937

The effect of pH value and pressure cooking on the mashing of corn.

Chester E. Brown
University of Louisville

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The Effect of pH Value and Pressure Cooking on the Mashing of Corn.

A Thesis
Submitted to the Faculty
Of the Graduate School
In Partial Fulfillment of the
Requirement for the Degree of
Master of Chemical Engineering.

Department of Chemical Engineering
By
Chester E. Brown

1937.
Chester E. Brown

The Effect of pH Value and Pressure Cooking on the Mashing of Corn

Director: ____________________________

Approved by Reading Committee ____________________________

Date: May 27, 1937
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ACKNOWLEDGMENT.
Acknowledgment.

The author wishes to express his appreciation for the helpful suggestions given by Doctor R. C. Ernest who directed this research, and for the assistance given by Miss Mattingly and Mr. Herin who worked with him in these experiments.

The author is indebted to Mr. F. M. Shipman, of the Brown-Forman Distillery Company for the grain used in these experiments, and for his helpful suggestions.
INTRODUCTION.
Introduction.

The object of this thesis is to determine the optimum pH for the diastase conversion of a corn mash and the affect of pressure cooking the corn prior to mashing.

Chemically, the process of converting the corn starch into sugar is hydrolysis. This may be carried out either by the action of the enzyme, diastase, or by boiling with dilute acid or by roasting. The first method is used almost exclusively in the fermentation industry of the United States, and for this reason the experimental work was confined to the diastase conversion.

The corn used is typical of the 1936 crop of corn sold to Kentucky distilleries. An analysis is included in the data. The soluble extract obtained by the treatment of this year's corn is almost as great as that obtained from crops of previous years. However, the extract contains a smaller percent of reducing sugar causing low alcoholic yields when fermented.

The pH used varied from 1.3 to 6.13 while the pressures studied were from atmospheric to 50 lbs. gage. The percent of reducing sugars and the total soluble extract were determined under each condition.
HISTORICAL

Chapter I.
History.

Many scientists have experimented with starch, but to date, even its structural formula remains uncertain. In 1872 C. O'Sullivan showed that the products resulting from the action of diastase on starch are maltose and dextrin; as the temperature of conversion is raised above 63°C, the proportion of maltose in the product decreases. According to Brown, Heron, and Morris (1879): When the temperature of conversion is at any temperature between 25°C - 60°C malt extract converts starch paste into 80.9 parts maltose and 19.1 parts dextrin.

Marquenne and Roux (1905) state that starch is composed of two substances, the envelope being of Amylopectin and interior of the granule Amylose. They obtained Amylose both by reversion and by heating starch with water under pressure and then cooling. They also state that this gives no coloration with iodine in the solid state, is not readily attacked by diastase, and is scarcely soluble in water at 120°C. When, however, the Amylose is heated under pressure to 150°C it dissolves, gives a blue color with iodine, and can be completely converted into maltose by malt extract.

The effect of cold concentrated and fuming HCl on starch was studied by Daisch (1914). He states that the acid converts starch into dextrins, maltose, and d-glucose.
In 1916 Davis put forward the idea that the enzyme diastase converts starch into dextrins, and maltose; and another enzyme - maltase - converts maltose into d-glucose.

Alsbert (1926) in his studies of starch, showed that when starch is heated with water the granules swell. Upon examining these swollen granules under the microscope, he found them to be of a sac-like structure filled with a liquid. When tannin was allowed to diffuse into the swollen granule, a strong Brownian movement could be detected under the microscope.

F. Emslander (1929) showed that the artificial acidification of mash and worts may greatly improve the stability of the beer, but often produces an "empty" and astringent flavor. The higher the litmus acidity that composes the salt-forming proteins, in proportion to the total acidity (with phenolphthalein), the fuller is the flavor of the beer.

Using the official German Congress method, Windisch, Kolbach, and Benedek (1929) found that at high temperatures the reaction in the mash tub should be less acid than at lower temperatures in order to secure the maximum of fermentable extract.

Windisch, Kolbach, and Schild (1932) concluded from their experiments that the rate of starch conversion within
the particular temperature range (up to 75°) increases with the rise in temperature. For the formation of maltose, there is a pronounced temperature optimum that varies somewhat according to the duration of mashing. The fermentable extract expressed as a percentage of the total extract attains its maximum at 63 - 64°. A prolonged mashing period increases the maltose. Evidence was found for the inhibitive action of lactates upon conversion of starch. For a prolonged mashing period, decrease in mash concentration lowers the proportion of fermentable sugars in the extract formed; probably because of the inactivity of the enzymes in thin mash.

A study of the influence of temperature and pH upon the formation of soluble noncoagulable N was made by Kolbach' (1932). He concluded that the pH optimum is about 4.3 - 4.7 at 50° and shifts toward the alkaline range at higher temperature. In 1936 experimenting at high temperatures, he disproved the idea that the enzyme, catalysing the first stages of conversion was more thermostable than the enzyme which completed the saccharification.
THEORY.

Chapter 2.
Theory.

When starch is heated with water to a temperature of about 70°C, the mixture begins to thicken or become pasty. Albert's investigating this paste under the microscope found that the granules swell but do not ordinarily burst even when heated to boiling. If, however, the paste is heated to still higher temperatures, the granules burst; and the sac-like cell wall, or starch cellulose, can be filtered from the solution. Since the cell wall seems to protect the granules and prevent the starch from reacting, it is very desirable to burst as many of the cell walls as possible. This is the purpose of pressure cooking the corn prior to conversion.

The enzyme diastase hydrolyses the starch to dextrins and then to maltose. The enzyme maltase then brings about the conversion of maltose to glucose. Enzymes apparently have many properties of lyophilic colloids. Therefore, we can expect the hydrogen ion concentration to have an influence on their activity.

Although the structural formula of starch has not yet been determined, the various degradation products have been given names, and the following scheme is representative of the course of hydrolysis.

\[
\text{Starch} \rightarrow \text{Amylodextrin} \rightarrow \text{Maltose} \rightarrow \text{Glucose}
\]
The dextrins can be distinguished from one another by the color they give with iodine. Amylodextrins give a blue, erythrodextrins a purple, and achroodextrin a red-brown. Stocks' suggests that the diastase simply peptizes the starch granules, instead of forming a series of compounds. The whole series of color reactions with iodine is a colloidal phenomenon depending upon the degree of dispersion—red for the smaller size and blue to black for the larger.

When maltose is being converted into dextrose (the temperature remaining constant), the velocity of the reaction is proportional to the concentration of maltose present at that instant since the water used does not appreciably alter the water concentration and it may, therefore, be assumed constant. The hydrolysis of maltose into dextrose is known as a "process of the first order" and may, be expressed mathematically by the differential equation

\[ \frac{dC}{dt} = KC \]  \[ C = Ke^{kt} \quad (1) \]

where \( C \) is the concentration of maltose at time \( t \), and \( K \) is the proportionality constant. If we let \( C_0 \) = the concentration of maltose at the start, then

\[ \int_{C_0}^{C} \frac{dC}{C} = \int_{0}^{t} Ke^{kt} \quad (2) \]
Integration gives

\[
\log (e) \frac{C}{C_0} = Kt \quad (3)
\]

If the value of \( C = C_1 \) at sometime \( t_1 \), then

\[
\log (e) \frac{C_1}{C_o} = Kt_1 \quad (4)
\]

or

\[
\log \frac{C}{C_o} = \log \frac{C_1}{C_o} \quad (5)
\]

\[
\log \frac{C}{C_o} = \log \left( \frac{C_1}{C_o} \right) \cdot t \quad (6)
\]

Therefore,

\[
\frac{C}{C_o} = \left( \frac{C_1}{C_o} \right)^{\frac{t}{t_1}} \quad (7)
\]

or \( C = C_o \left( \frac{C_1}{C_o} \right)^{\frac{t}{t_1}} \)

gives the concentration of maltose at anytime \( t \): having
given the concentration at the beginning \( C_o \), and the concent-
tration \( C_1 \) at some later time \( t_1 \).

From the preceding statements, we might expect the
degree of hydrolysis to be dependent upon four things:
1. The temperature at which the hydrolysis is taking place.
2. The hydrogen ion concentration of the solution.
3. The condition of the starch cells.
4. The length of time the hydrolysis has proceeded.
The optimum temperature for the hydrolysis, as determined by Windisch, Kolbach, and Schild, is 63° - 64° C.

It is a well established fact that a sour mash gives a higher alcoholic yield, but, up to a temperature of 70° C, the acidity should be decreased as the temperature of conversion is increased. So, we can expect the maximum conversion to take place in a slightly acid solution.

It should be noted that since two enzymes are responsible for the conversion of starch into dextrose, they will not necessarily have the same optimum pH. However, the rate of formation of dextrose is proportional to the amount of maltose present; and if the enzyme maltase has an optimum at a pH value, which is not very suitable for the enzyme diastase, the optimum pH for the production of dextrose would not necessarily be the optimum for the enzyme maltase when the two enzymes are present in the same solution, because at its optimum pH the maltose would have very little maltose to act upon. From this we can expect the optimum pH for the production of maltose and dextrose to be fairly close together or at least closer together than they would be if studied separately.
Starch cannot be completely converted into dextrose, but amylose, the substance on the interior of the starch granule, undergoes complete hydrolysis. Therefore, if the amylose can be separated from the protection of the amylopectin it can be completely converted into dextrose. Heating the corn to high temperatures seems to decrease the protection power of the amylopectin and to allow the diastase to attack the amylose. The amount of the starch that is converted depends upon the length of time that the hydrolysis has been going on as well as the condition of the starch granules. The optimal pressure and time of cooking should not be expected to remain constant if the time of mashing is varied. If the time of mashing is held constant, however, we should be able to observe marked differences in the amount of conversion that takes place in pastes that have been cooked at different pressures and for various lengths of time.
APPARATUS

Chapter 3.
Apparatus

Apparatus for pH Determination.

A 1000 cc beaker was used to heat the mash which was agitated throughout the run by means of a small electric stirrer.

The mash was filtered in a Buchner funnel with the aid of a filter cell and a suction filter.

The pH value of the water was determined by a quinhydrone electrode. This apparatus consists of a Leeds and Northrup Quinhydrone potentiometer, a saturated calomel, and a platinum electrode.

The pH value of the mash was determined by a glass electrode, consisting of a Leeds and Northrup Portable potentiometer-electrometer a saturated calomel, and a glass electrode.

Apparatus for Pressure.

The cooking was done in a ten gallon autoclave equipped with an agitator. The temperature was controlled by passing steam or water through the jacket around the centoclave. The fermentation took place in a two gallon crock, and distilled in a two gallon copper still directly connected to a copper condenser by a brass pipe. The condenser was fitted with ten feet of block-tin coil and connections for the circulation of cooling water.
The condensate was run into a 1000 cc erlenmeyer flask, and the specific gravity was determined with a Westphale balance.
PROCEDURE

Chapter 4.
Procedure.

In determining the optimum pH the charge was as follows:

157.5 grams of ground corn
17.5 grams of malt
700.0 cc of distilled water

The distilled water was boiled to expel the CO₂ and a calculated quantity of 0.1N HCl or 0.1N NaOH added to give approximately the desired pH. The exact pH of this solution was then determined by a quinhydrone electrode.

The water was heated in a beaker until it began to boil, and the corn was added with the constant stirring. The beaker was placed in a hot water bath and a temperature of 90 - 95°C was maintained for 30 minutes. During this time the mixture was continuously agitated.

The paste was cooled to 65°C, the malt added, and the temperature kept at 62 - 65 for 30 minutes. After 15 minutes of cooling in a cold water bath, the soluble dextrins and dextrose were separated from the insoluble material by filtration.

During run No. 8 the pH value was determined every ten minutes after the corn was added and every ten minutes during mashing.
The charge for the pressure cooking was as follows:

- 8.25 lbs. corn
- 0.92 lbs. malt
- 36.5 lbs. city water

Thirty-one and five tenths lbs. of city water was poured into the cooker, the agitator started and then the corn added. The cap was screwed on, the steam turned into the jacket, and the corn was cooked at different temperatures for various lengths of time. After cooking was completed the steam was shut off and cold water run through the jacket until the temperature of the mixture reached 70°C. At this point the malt mixed with 5 lbs. of water was added and a temperature of 62 - 65°C was maintained for thirty minutes. The mash was cooled, for five minutes, filtered, and the amounts of dextrose and dextrin present were determined.

One gallon of the unfiltered mash from runs No. 23 and No. 26 were fermented for seventy-two hours and then distilled. The percent alcohol was determined from its specific gravity.

**Dextrose Determination.**

Twenty-five cc of the filtered solution was diluted, with distilled water, to 250 cc, and the resulting solution titrated against 25 cc of standard Benedict's solution. This indicated the amount of dextrose or reducing sugar present.
Total Dissolved Carbohydrates Determination.

Another 25 cc of the sugar solution was diluted, with distilled water, to 100 cc and refluxed for thirty minutes with 3 cc of concentrated HCl. This converted the dextrins to dextrose. This dextrose solution was then diluted to 500 cc with distilled water and titrated against 25 cc of Benedict's solution. This indicated the amount of dextrose and other carbohydrates originally present.

Procedure for Grain Analysis.

The grain was analyzed according to the procedure given in Leach's "Food Inspection and Analysis".

Moisture Determination.

A two gram sample was weighed in a flat dish and heated in an oven at 100° for six hours. The sample was cooled in a desiccator and weighed.

Determination of Percent Fat.

The residue for the moisture determination was extracted for sixteen hours with anhydrous alcohol-free ether in a continuous extractor and the extract dried to constant weight at 100°.
Total Nitrogen.

A one gram sample was introduced into a Kjeldahl flask and 10 grams of K₂SO₄ and 20 cc of concentrated H₂SO₄ added. The flask was heated gently below the boiling point until frothing ceased and then boiled until colorless. The solution was cooled and diluted with 100 cc of water, then 25 cc of saturated NaOH was added and the mixture distilled into 0.10N H₂SO₄. The excess H₂SO₄ was titrated with 0.10N NaOH.

Ash Determination.

A porcelain crucible was ignited, cooled, and weighed. Two grams of the grain sample was brushed into the crucible and burned at a low temperature with a meaker burner. The crucible was kept at a dull redness until the carbon was consumed and the ash became nearly white. The crucible was cooled in a desicator and weighed.

Determination of Sugar, Dextrin, Starch, and Cellulose.

One hundred grams of corn was refluxed with 500 cc of 95% alcohol for three hours and filtered. The filtrate was evaporated nearly to dryness and then was taken up in a little water. The solution was then inverted to boiling with 2 cc of concentrated HCl and the sugar determined with Benedict's solution.
The residue from the alcoholic treatment was extracted from eighteen hours with 500 cc of cold distilled H₂O. The mixture was filtered and the filtrate concentrated to 100 cc. To this 2 cc of concentrated HCl was added, and the solution refluxed for one-half hour. The dextrin was determined by Benedict's solution.

The residue from the water extraction was dried in an oven and its quantitative relation to the original sample determined. Two grams were then accurately weighed and subjected to acid conversion. The mixture was filtered and the starch determined by Benedict's solution.

The residue from the acid hydrolysis was boiled for one-half hour with 200 cc of 1.25% NaOH, filtered, dried, and weighed.

Preparation of Benedict's Solution.

1. CuSO₄ (Crystal) 18 grams
2. Na₂ CO₃ (Monohydrate) 150 grams
3. Sodium Citrate 200 grams
4. KCnS 125 grams
5. K₄ Fe (CN)₆ (5%Sol) 5 cc

(a) Dissolve by heat - 2, 3, and 4 in 800 cc distilled water.
(b) Dissolve 1 in 100 cc of H₂O and add to (a) with stirring - add 5, cool, and dilute to 1000 cc.

Twenty-five cc of this solution was titrated against a standard sugar solution to determine its strength.
DATA AND RESULTS.

Chapter 5.
Data and Results.

The effect of varying the pH on the conversion of starch is shown in tables 1 - 4 inclusive and the results are plotted on page 42.

There is no change in the amount of conversion as the pH raises from 3.16 to 5. The amount of carbohydrates in solution increases rapidly when the pH is raised above 5.1. They go from an almost constant value, between a pH of 3.16 - 5, to a maximum at a pH of 5.5. The soluble carbohydrates changing from 10.7 to 11.55g per 100cc - the dextrose from 5.10 to 5.65, and the dextrin from 5.65 to 5.90. As the pH value rises from 5.6 to 6.4 there is a rapid decrease in the amount of soluble carbohydrates, while the amount of dextrose decreases very slowly.

The amount of dextrin left in solution reaches a minimum of 4.59g per 100 cc at a pH of 6.4 while the amount of dextrose is 5.31. When the pH rises above 6.4 the amount of dextrose decreases much more rapidly than the amount of soluble carbohydrates until at a pH of 7.0 the amount of dextrin (4.99g per 100cc) is slightly higher than that of dextrose (4.83g per 100cc). At a pH of 8.13 there are 2.69g per 100cc of soluble carbohydrates while the amount of dextrose has decreased to 2.3g per 100cc leaving 5.89 grams per 100cc of dextrin.
The effect of pressure-cooking the corn prior to mashing is shown in the Tables 5 to 9, and the results are plotted on pages 43 - 52.

The soluble carbohydrates produced is a straight line function of the length of time that the corn has been cooked, within the range of pressures studied. The concentration varied from 8.25g per 100cc (Atmos. - 10 min.) to 12.6g per 100cc (50 lbs. - 40 min.).

The dextrin and dextrose, when cooked from 10 to 40 minutes at atmospheric pressure varied from 2.23 to 5.62 and from 1.19 to 3.63 respectively, and did not show any regularity.

When cooked at pressures of 15, 30, and 40 lbs. per sq. in. the dextrose approaches a maximum of 3.55, 4.32, 5.70 at the respective pressures. The dextrin approaches a straight line function of time after 20 - 25 minutes of cooking at 15, 30, and 40 lbs. per sq. in.

The advantage of mashing for 60 minutes instead of 30 is shown in Table 10. The amount of dextrose was increased from 3.30 to 4.53 and from 2.91 to 4.12g per 100cc, while the soluble carbohydrates was increased from 5.5 to 8.05 and from 5.5 to 733g per 100cc in the two runs studied.
Table 1.

Effect of Varying the pH from 3.16 to 5.87 on the Conversion of Starch.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quinhydrone Potentiometer Reading</td>
<td>-266m.v.</td>
<td>-214m.v.</td>
<td>-121m.v.</td>
<td>-106m.v.</td>
</tr>
<tr>
<td>pH Value</td>
<td>3.16</td>
<td>4.04</td>
<td>5.61</td>
<td>5.87</td>
</tr>
<tr>
<td>cc of Dextrose Solution</td>
<td>10.11</td>
<td>9.79</td>
<td>9.90</td>
<td>10.1</td>
</tr>
<tr>
<td>cc of Soluble Carbohydrate Solution</td>
<td>9.40</td>
<td>9.30</td>
<td>9.70</td>
<td>8.25</td>
</tr>
<tr>
<td>Strength of Benedict’s Solution</td>
<td>.0504g</td>
<td>.0504g</td>
<td>.0560</td>
<td>.04617</td>
</tr>
<tr>
<td>Carbohydrates in 100cc of Mash. (Expressed as grams of C₆H₁₀O₅)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Run No.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total Carbohydrates</td>
<td>14.10</td>
<td>14.10</td>
<td>14.10</td>
<td>14.10</td>
</tr>
<tr>
<td>Soluble Carbohydrates</td>
<td>10.70</td>
<td>10.75</td>
<td>11.55</td>
<td>11.20</td>
</tr>
<tr>
<td>Converted to Dextrose</td>
<td>5.00</td>
<td>5.15</td>
<td>5.65</td>
<td>5.48</td>
</tr>
<tr>
<td>Converted to Dextrin</td>
<td>5.70</td>
<td>5.61</td>
<td>5.90</td>
<td>5.72</td>
</tr>
</tbody>
</table>
Table 2.

Effect of varying the pH from 6.4 to 8.13 on the Conversion of Starch.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>5</th>
<th>6</th>
<th>7</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quinhydrone Rotentiometer</td>
<td>-78m.v.</td>
<td>-395m.v.</td>
<td>+23m.v.</td>
</tr>
<tr>
<td>Reading</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>pH Value</td>
<td>6.40</td>
<td>7.00</td>
<td>8.13</td>
</tr>
<tr>
<td>cc of Dextrose Solution</td>
<td>8.65</td>
<td>11.6</td>
<td>20.00</td>
</tr>
<tr>
<td>cc of Soluble Carbohydrate Solution</td>
<td>9.35</td>
<td>11.4</td>
<td>12.9</td>
</tr>
<tr>
<td>Strength of Benedict's Solution</td>
<td>0.04617</td>
<td>0.0560</td>
<td>0.0010</td>
</tr>
<tr>
<td>Carbohydrates in 100cc of Mash.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(Expressed as grams of C_6H_{12}O_{6})</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Run No.</td>
<td>5</td>
<td>6</td>
<td>7</td>
</tr>
<tr>
<td>Total Carbohydrates</td>
<td>14.10</td>
<td>14.10</td>
<td>14.10</td>
</tr>
<tr>
<td>Soluble Carbohydrates</td>
<td>9.30</td>
<td>9.92</td>
<td>8.69</td>
</tr>
<tr>
<td>Converted to Dextrose</td>
<td>5.31</td>
<td>4.83</td>
<td>2.90</td>
</tr>
<tr>
<td>Converted to Dextrin</td>
<td>4.59</td>
<td>4.99</td>
<td>5.89</td>
</tr>
</tbody>
</table>
Table 3.

Changes in pH taking place during mashing and the conversion obtained using water with a pH at 5.1.

(Run No.8  pH = 5.1)

Quinhydrone potentiometer reading = -151 m.v. (25°C).

<table>
<thead>
<tr>
<th>Time</th>
<th>Glass Electrode Potentiometer</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>At Start</td>
<td>-0.155</td>
<td>5.1</td>
</tr>
<tr>
<td>30 minutes after corn was added</td>
<td>-0.170</td>
<td>5.30</td>
</tr>
<tr>
<td>10 minutes after malt was added</td>
<td>-0.150</td>
<td>5.70</td>
</tr>
<tr>
<td>20 minutes after malt was added</td>
<td>-0.150</td>
<td>5.70</td>
</tr>
<tr>
<td>30 minutes after malt was added</td>
<td>-0.150</td>
<td>5.70</td>
</tr>
</tbody>
</table>

Dextrose Determination.

25cc of filtered mash diluted to 250cc.

9.35cc = 25cc Benedict’s solution.

Soluble Carbohydrates.

25cc refluxed with HCl and diluted to 500.

9.30cc = 25cc Benedict’s solution.

Carbohydrates in 100cc of Mash.

(Expressed as grams of C₆H₁₂O₆)

Total carbohydrates = 14.10
Soluble carbohydrates = 10.76
Converted to dextrose = 5.03
Converted to Dextrin = 5.73

* 25cc of this Benedict’s solution = 50.01 mg C₆H₁₂O₆.
Table 4.

Conversion obtained using water
with a pH of 1.3.

(Run No. 9     pH = 1.3)

Quinhydrone potentiometer reading = -376m.v. (25°C).

Dextrose Determination.

25cc of solution diluted to 250cc.

93.2cc = 25cc of Benedict's solution.

The solution was not filterable and the soluble carbohydrates could not be determined. The dextrose was determined in the presence of the starch.

There was 0.644 grams starch converted to dextrose per 100cc of solution.
Table 5.
Starch Conversion at Atmospheric Pressure.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>23</th>
<th>24</th>
<th>25</th>
<th>26</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time of Cooking (Minutes)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>40</td>
<td>30</td>
<td>20</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td>cc of Dextrose</td>
<td>13.21</td>
<td>16.00</td>
<td>16.21</td>
<td>28.50</td>
</tr>
<tr>
<td>cc of Soluble Carbohydrates</td>
<td>11.60</td>
<td>13.70</td>
<td>17.20</td>
<td>24.50</td>
</tr>
<tr>
<td>Strength of Benedict's Solution</td>
<td>.0480g</td>
<td>.0480g</td>
<td>.0480g</td>
<td>.0480g</td>
</tr>
<tr>
<td>Carbohydrates in 100cc of Mash.</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(Expressed as grams of C₆H₁₂O₆)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Run No.</td>
<td>23</td>
<td>24</td>
<td>25</td>
<td>26</td>
</tr>
<tr>
<td>Total Carbohydrates</td>
<td>14.1</td>
<td>14.1</td>
<td>14.1</td>
<td>14.1</td>
</tr>
<tr>
<td>Soluble Carbohydrates</td>
<td>8.25</td>
<td>7.00</td>
<td>5.50</td>
<td>3.92</td>
</tr>
<tr>
<td>Converted to Dextrose</td>
<td>3.63</td>
<td>4.00</td>
<td>2.91</td>
<td>1.69</td>
</tr>
<tr>
<td>Converted to Dextrin</td>
<td>5.62</td>
<td>3.00</td>
<td>2.59</td>
<td>2.23</td>
</tr>
</tbody>
</table>
Table 6
Starch Conversion at Pressure 15 lbs. per square inch.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>19</th>
<th>20</th>
<th>21</th>
<th>22</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time of Cooking (Minutes)</td>
<td>40</td>
<td>30</td>
<td>20</td>
<td>10</td>
</tr>
<tr>
<td>cc of Dextrose</td>
<td>13.5</td>
<td>13.4</td>
<td>14.50</td>
<td>19.2</td>
</tr>
<tr>
<td>cc of Soluble Carbohydrates</td>
<td>10.5</td>
<td>11.8</td>
<td>14.75</td>
<td>17.6</td>
</tr>
<tr>
<td>Strength of Benedict’s Solution</td>
<td>0.0480g</td>
<td>0.0462g</td>
<td>0.0490g</td>
<td>0.0480g</td>
</tr>
<tr>
<td>Carbohydrates in 100cc of Mash. (Expressed as grams of C₆H₁₀O₅)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Run No.</td>
<td>19</td>
<td>20</td>
<td>21</td>
<td>22</td>
</tr>
<tr>
<td>Total Carbohydrates</td>
<td>14.1</td>
<td>14.1</td>
<td>14.1</td>
<td>14.1</td>
</tr>
<tr>
<td>Soluble Carbohydrates</td>
<td>9.15</td>
<td>7.83</td>
<td>6.50</td>
<td>5.45</td>
</tr>
<tr>
<td>Converted to Dextrose</td>
<td>3.55</td>
<td>3.45</td>
<td>3.30</td>
<td>2.50</td>
</tr>
<tr>
<td>Converted to Dextrin</td>
<td>5.60</td>
<td>4.38</td>
<td>3.20</td>
<td>2.95</td>
</tr>
</tbody>
</table>
## Table 7

Starch Conversion at Pressure 30 lbs. per square inch.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>15</th>
<th>16</th>
<th>17</th>
<th>18</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time of Cooking (Minutes)</td>
<td>40</td>
<td>30</td>
<td>20</td>
<td>15</td>
</tr>
<tr>
<td>cc of Dextrose</td>
<td>10.7</td>
<td>10.65</td>
<td>12.80</td>
<td>14.80</td>
</tr>
<tr>
<td>cc of Soluble Carbohydrates</td>
<td>8.81</td>
<td>10.20</td>
<td>12.00</td>
<td>14.80</td>
</tr>
<tr>
<td>Strength of Benedicta Solution</td>
<td>0.0460g</td>
<td>0.0460g</td>
<td>0.0480g</td>
<td>0.0480g</td>
</tr>
</tbody>
</table>

Carbohydrates in 100cc of Mash.

(Expressed as grams of C\(_6\)H\(_{12}\)O\(_6\))

<table>
<thead>
<tr>
<th>Run No.</th>
<th>15</th>
<th>16</th>
<th>17</th>
<th>18</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total Carbohydrates</td>
<td>14.1</td>
<td>14.1</td>
<td>14.1</td>
<td>14.1</td>
</tr>
<tr>
<td>Soluble Carbohydrates</td>
<td>10.50</td>
<td>9.06</td>
<td>7.49</td>
<td>6.48</td>
</tr>
<tr>
<td>Converted to Dextrose</td>
<td>4.32</td>
<td>4.35</td>
<td>4.00</td>
<td>3.24</td>
</tr>
<tr>
<td>Converted to Dextrin</td>
<td>6.18</td>
<td>4.71</td>
<td>3.49</td>
<td>3.24</td>
</tr>
</tbody>
</table>
Table 8

Starch Conversion at Pressure 40 lbs. per square inch.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>11</th>
<th>12</th>
<th>13</th>
<th>14</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time of Cooking (Minutes)</td>
<td>40</td>
<td>30</td>
<td>20</td>
<td>10</td>
</tr>
<tr>
<td>cc of Dextrose</td>
<td>8.80</td>
<td>9.2</td>
<td>9.35</td>
<td>15.6</td>
</tr>
<tr>
<td>cc of Soluble carbohydrates</td>
<td>8.10</td>
<td>9.05</td>
<td>10.00</td>
<td>11.7</td>
</tr>
<tr>
<td>Strength of Benedict's Solution</td>
<td>.0504g</td>
<td>.0504g</td>
<td>.0504g</td>
<td>.0504g</td>
</tr>
<tr>
<td>Carbohydrates in 100cc of Mash. (Expressed as grams of C(<em>6)H(</em>{12})O(_6))</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Run No.</td>
<td>11</td>
<td>12</td>
<td>13</td>
<td>14</td>
</tr>
<tr>
<td>Total Carbohydrates</td>
<td>14.1</td>
<td>14.1</td>
<td>14.1</td>
<td>14.1</td>
</tr>
<tr>
<td>Soluble Carbohydrates</td>
<td>12.35</td>
<td>11.10</td>
<td>10.00</td>
<td>8.55</td>
</tr>
<tr>
<td>Converted to Dextrose</td>
<td>5.70</td>
<td>5.47</td>
<td>5.35</td>
<td>3.21</td>
</tr>
<tr>
<td>Converted to Dextrin</td>
<td>6.65</td>
<td>5.63</td>
<td>4.65</td>
<td>5.34</td>
</tr>
</tbody>
</table>
Table 9
Starch Conversion at Pressure 50 lbs. per square inch.

Run No. 10 - Time of Cooking 40 minutes.

Dextrose Determination.

25cc of filtered mash diluted to 250cc
9.4 = 25cc of Benedict's solution

Soluble Carbohydrates.

25cc of filtered mash refluxed with HCl diluted to 500cc
8.9cc of this solution = 25cc of Benedict's solution

Carbohydrates in 100cc of Mash.
(Expressed as grams of C₄H₁₀O₅)

| Total carbohydrates | = 14.10 |
| Soluble carbohydrates | = 12.6 |
| Converted to dextrose | = 5.95 |
| Converted to dextrin | = 6.65 |

* 25cc of this Benedict's solution = 56.00mg C₄H₁₀O₅.
Table 10

Effect ofwashing for 60 minutes
on Conversion.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>21</th>
<th>25</th>
</tr>
</thead>
<tbody>
<tr>
<td>cc of Dextrose</td>
<td>10.60</td>
<td>11.65</td>
</tr>
<tr>
<td>cc of Soluble Carbohydrates</td>
<td>11.90</td>
<td>13.15</td>
</tr>
</tbody>
</table>

Strength of Benedict's Solution

<table>
<thead>
<tr>
<th>Run No.</th>
<th>21</th>
<th>25</th>
</tr>
</thead>
<tbody>
<tr>
<td>C₆H₁₂O₆</td>
<td>0.0480g</td>
<td>0.0480g</td>
</tr>
</tbody>
</table>

Carbohydrates in 100cc of Mash.
(Expressed in grams of C₆H₁₀O₅)

<table>
<thead>
<tr>
<th>Run No. 21</th>
<th>Run No. 25</th>
</tr>
</thead>
<tbody>
<tr>
<td>30 minutes</td>
<td>60 minutes</td>
</tr>
<tr>
<td>mashing</td>
<td>mashing</td>
</tr>
<tr>
<td>Soluble Carbohydrates</td>
<td>6.5</td>
</tr>
<tr>
<td>Converted to Dextrose</td>
<td>3.3</td>
</tr>
<tr>
<td>Converted to Dextrin</td>
<td>3.2</td>
</tr>
</tbody>
</table>
Table 11.

Fermentation of Mashes Containing Different Quantities of Dextrose.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Grams of Dextrose</th>
<th>Grams of Dextrine</th>
<th>cc of Distillate</th>
<th>Specific Gravity</th>
<th>cc of 100 proof Alcohol</th>
</tr>
</thead>
<tbody>
<tr>
<td>26</td>
<td>1.69</td>
<td>2.23</td>
<td>412</td>
<td>0.961</td>
<td>260</td>
</tr>
<tr>
<td>23</td>
<td>3.63</td>
<td>5.62</td>
<td>492</td>
<td>0.970</td>
<td>337.0</td>
</tr>
<tr>
<td></td>
<td>Corn</td>
<td>Malt</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>------------------</td>
<td>--------</td>
<td>--------</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Water</td>
<td>12.42</td>
<td>5.24</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sugar, Gum and Dextrin</td>
<td>6.32</td>
<td>18.10</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Starch</td>
<td>59.78</td>
<td>47.22</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cellulose</td>
<td>4.69</td>
<td>12.30</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ash</td>
<td>1.23</td>
<td>2.52</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Protein</td>
<td>10.41</td>
<td>13.70</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fat</td>
<td>5.15</td>
<td>1.92</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Curve No. 0

Soluble Carbohydrates

Dextrin

Dextrose

Conversion (Grams C₆H₁₂O₆)

pH Value

pH Value
Curve No. 1
C₆H₁₀O₅ Conversion
At Atmospheric Pressure

Conversion (Grams of C₆H₁₀O₅)

Time of Cooking (Minutes)

Soluble Carbohydrates
Dextrin
Dextrose
Curve No. 2

C₆H₁₀O₅ Conversion

At

15 * Pressure

Conversion (Grams of C₆H₁₀O₅)

Soluble Carbohydrates

Dextrin

Dextrose

Time of Cooking (Minutes)
Curve No. 3

$C_6H_{10}O_5$ Conversion

At

30# Pressure

Conversion (grams of $C_6H_{10}O_5$)

Time of Cooking (Minutes)
Curve No. 4

$C_6H_{12}O_6$ Conversion at 40° Pressure

- Soluble Carbohydrates
- Dextrose
- Dextrin

Time of Cooking (Minutes)
Curve No. 5

C₆H₁₀O₅ Conversion During 10 Minute Cooking

Conversion (Grams of C₆H₁₀O₅)

Gage Pressure of Cooking (Lbs. per Sq. In.)
Curve No. 6

C₆H₁₀O₅ Conversion during 20 Minute Cooking

Conversion (Grams of C₆H₁₀O₅)

Pressure of Cooking (Lbs. per Sq. In. Gage)
Curve No. 7

C₆H₁₀O₅ Conversion during 30 Minute Cooking

Conversion (Grams of C₆H₁₀O₅)

Gas Pressure of Cooking (Lbs./Sq. In.)
Curve No. 8

C₆H₁₀O₅ Conversion During 40 Minute Cooking

Conversion (grams of C₆H₁₀O₅)

Gaga Pressure of Cooking (Lbs./Sq.In.)
CURVE No. 9

C₆H₁₀O₅ CONVERSION AT VARIABLE Pressures

CONVERSION (Grams C₆H₁₀O₅)

TIME of COOKING (MINUTES)
Sample Calculation.

Run 1.

\[
\text{pH Value} = \frac{456 + E}{0.0591}; \quad E = -266 \text{ m.v.}
\]

\[
= \frac{0.456 + (-266)}{0.0591} = 3.16
\]

Soluble Carbohydrates = \text{Strength of Benedict's Sol.} \quad (500)
\[
\text{cc of Solution} \times (25) \times 100
\]

\[
= \frac{0.0504}{9.40} \times 2,000 = 10.70 \text{g per 100cc.}
\]

Dextrose = \text{Strength of Benedict's Sol.} \quad (250)
\[
\text{cc of Solution} \times (25) \times 100
\]

\[
= \frac{0.0504}{10.11} \times 1,000 = 5.00
\]

Total Carbohydrates = \text{Carbohydrates in Com.} = \text{Carb. in Malt}
\[
\text{in 100cc} \quad \text{Final Volume} \times 100
\]

\[
= 157.5 \times 66.1\% + 17.5 \times 65.3\% \times \frac{1,065}{700 + 157.5 + 17.5} \times 100
\]

\[
= 14.10
\]
INTERPRETATION OF RESULTS.

Chapter 6.
Interpretation of Results
Obtained by Varying the pH Values.

The activity of both enzymes, maltase and diastase, increases as the acidity of the mash is increased from a slightly basic solution (pH=8) to a neutral one. The activity of the enzyme maltase increases more rapidly than that of diastase, as shown by the decrease in the amount of dextrin present.

The activity of the enzyme diastase begins to increase very rapidly after a pH of 6.5 and reaches its optimum at a pH of about 5.5. At this pH the concentration of dextrose is also at a maximum. The optimum pH for the enzyme maltase is at a somewhat higher value, or at a pH value of 6.4 the concentration of the dextrose approaches its maximum value even though the concentration of dextrin, from which dextrose is formed, is at a minimum.

When the pH value of the water is approximately one, the production of dextrose has nearly ceased. This indicates that the increase in alcoholic yield obtained by using sour mash is due to the increased activity of the enzymes in acid solutions at low temperatures, rather than the combined effect of the enzymes and acid hydrolysis.

The optimum pH of the water lies between 5.4 and 5.8. The pH value of the mash remains constant after the malt has been well mixed with the paste.
Interpretation of Results Obtained
By Pressure Cooking the Corn.

When corn is cooked at constant pressure for varied lengths of time the amount of starch that can be converted, by allowing diastase to act upon the paste for thirty minutes, is a straight line function of the time the corn has been cooked, so long as cooking has not proceeded longer that the time required to convert all of the starch. On the other hand the dextrose reaches a maximum after about twenty-five minutes of cooking at the pressures studied, and seems to be limited by the activity of the enzyme.

Considering time as a constant, the amount of starch converted and the amount of dextrose produced increase very rapidly up to a pressure of about 40 lbs. per square inch. Increasing the pressure from 30 - 40 lbs. per square inch, the cooking being allowed to proceed for twenty minutes in each case, brought about a 37.5 percent increase in the concentration of dextrose. When the pressure was increased to 50 lbs. per square inch and cooked for forty minutes the concentration of dextrose increased only 9.3 percent above the amount obtained by cooking the corn for only twenty minutes at 40 lbs. per square inch. Therefore, pressure cooking the corn at about 40 lbs. per square inch for twenty or twenty-five minutes would be most economical.
Allowing the action of the enzymes to continue for one hour gives a marked increase in dextrose production and starch conversion.

Fermentation and distillation of mashes containing the same amount of corn, but having undergone different degrees of conversion yield quantities of alcohol which are dependent upon the amount of sugar present in the mash.
BIBLIOGRAPHY.
Bibliography.


8. Bradley - Kentucky Whiskey 1994 Louisville Public Library (K-685.5; B - 811).


Effect of pH on Starch Conversion

Curve No.

Concentration (Grams C₆H₁₀O₅)

pH Value

Soluble Carbohydrates

Dextrin

Dextrose
Curve No. 1

C₈H₁₀O₅ Conversion

Atmospheric Pressure

Conversion (Grams of C₈H₁₀O₅)

Time of Cooking (Minutes)
CURVE No. 2
C₆H₁₀O₅ CONVERSION
AT
15 # PRESSURE

CONVERSION (GRAMS of C₆H₁₀O₅)

TIME of COOKING (MINUTES)

Soluble Carbohydrates
Dextrin
Dextrose
Curve No. 3

$C_6H_{10}O_5$ Conversion

at 30# Pressure

Time of Cooking (Minutes)
Curve No. 4

C₆H₁₀O₅ Conversion at 40# Pressure

(Conversion of C₆H₁₀O₅)

Soluble Carbohydrates

Dextrose

Dextrin

Time of Cooking (Minutes)
Curve No. 5

C$_6$H$_{10}$O$_5$ Conversion during 10 Minute Cooking

Conversion (Grams of C$_6$H$_{10}$O$_5$)

Gage Pressure of Cooking (Lbs. per Sq. In.)

- Soluble Carbohydrates
- Dextrin
- Dextrose
Curve No. 6

$C_6H_{10}O_5$ Conversion during 20 Minute Cooking

Soluble Carbohydrates

Dextrose

Dextrin

Conversion (Grims of $C_6H_{10}O_5$)

Pressure of Cooking (Lbs. per Sq. In. Gage)
Curve No. 7

C₆H₁₀O₅ Conversion during 30 Minute Cooking

Gage Pressure of Cooking (Lbs./Sq. In.)

- Soluble Carbohydrates
- Dextrin
- Dextrose
Curve No. 8

\( C_6H_{10}O_5 \) Conversion during 40 Minute Cooking

Conversion (Grams of \( C_6H_{10}O_5 \))

Gage Pressure of Cooking (Lbs./Sq.In.)

Soluble Carbohydrates

Dextrin

Dextrose
Curve No. 9

C₆H₁₀O₅ Conversion at Variable Pressures

Conversion (Grams C₆H₁₀O₅)

Time of Cooking (Minutes)
Curve No. 10

Conversion of C₆H₁₀O₅ (Grams)

40°/Sq. In. Gage
30°/Sq. In. Gage
15°/Sq. In. Gage

Atmospheric

C₆H₁₀O₅ Conversion
To Dextrose

Time of Cooking (Minutes)